

NASA TECHNICAL
MEMORANDUM

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MATERIALS PROCESSING IN SPACE
PROGRAM TASKS

Compiled by E. Pentecost
Space Sciences Laboratory

1981

NASA



*George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama*

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NASA TECHNICAL MEMORANDUM

I. INTRODUCTION

The Materials Processing in Space program is directed toward research in the science and technology of processing materials under conditions of low gravity to provide a detailed examination of the constraints imposed by gravitational forces on Earth. The program is expected to lead, ultimately, to the development of new materials and processes in commercial applications adding to this nation's technological base. The research studies emphasize the selected materials and processes that will best elucidate the limitations due to gravity and demonstrate the enhanced sensitivity of control of processes that may be provided by the weightless environment in space. Primary effort will be devoted to a comprehensive study of the specific areas of research which revealed potential value in the initial investigations of the previous decade. Examples of previous process research include growth of crystals and directional solidification of metals in the quiescent conditions in which gravitational fluid flow is eliminated, containerless processing of reactive materials to eliminate reactions with the container and to provide geometrical control of the product, synthesis and separation of biological materials in weightlessness to reduce heat and mass transfer problems associated with sedimentation and buoyancy effects, identification of high vacuum characteristics associated with an orbiting wake shield, minimal knowledge of terrestrial processing methods.

Additional effort will be devoted to identifying the special requirements which drive the design of hardware to reduce the risk in future developments. Examples of current hardware studies are acoustic, electromagnetic and electrostatic containerless processing modules, and electrophoresis separation devices.

In addition to the basic research nature of the program, a lower level of effort is being expended on the business, a logistics and legal implication of rights of data and patents, control of materials, and division of responsibilities when NASA works with commercial ventures aimed at specific products. Examples of current materials research which might lead to commercialization include infrared detector crystals, inertial confinement fusion targets, electrolytes with dispersoids, aligned magnets, and ferromagnetic materials.

History

Materials Processing in Space was initiated with a few simple demonstrations of principles by astronauts on Apollo missions 14 and 16. These were followed by more extensively planned but limited experiments on Skylab in 1974. On Apollo-Soyuz in 1975, many of the experiments were repeated with other materials and

improved measurements. Also on that mission, electrophoresis experiments gave early indications of the possibility of improved separations of biological materials. In all, more than 50 flight experiments or demonstrations of fundamental effects of weightlessness were completed. Since the end of the Apollo program, the flight opportunities for researchers have been limited to ballistic rocket flights providing approximately 5 minutes of weightlessness during the coasting phase of flight. Approximately 30 experiments have been completed on Space Processing Applications Rockets (SPAR's).

A recent assessment of the program was made by a committee of the National Research Council on the Scientific and Technological Aspects of Materials Processing in Space (STAMPS). Recommendations stressed the need for more extensive ground-based research to serve as a support base for the evolution and assessment of investigations which would lead to a proper understanding of the role played by gravity in materials processes. Recourse to the weightless environment of space should be based primarily on the understanding and need in those specific cases identified from such a program. In addition, the first phase of the spaceflight program should be a demonstration of the new technology developed in the NASA program which should then be transferred to non-NASA entities for their use. The second phase, funded primarily by non-NASA users, should consist of a National Materials Laboratory in space to open the capabilities to all for a reasonable charge. Closer ties between the materials communities and NASA were recommended in the form of peer review of all proposals, both ground-based and spaceflight, and the periodic peer review of policies and plans. The Materials Processing in Space program has been restructured on the basis of these recommendations beginning with the earliest deliberations of the STAMPS Committee. An advisory committee has been formed to provide guidance in future program planning and policy making that is consistent with the spirit and principle of the STAMPS Committee recommendations.

Program Strategy

The current program emphasis on fundamental processing science and technology in selected areas will continue for the period of this plan as the Materials Processing in Space program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment. During this phase of the program, the development and demonstration of current space technology for materials processing will be transferred, as appropriate, to non-NASA users. In order to assist this process, a Commercial Space Processing Task Team has been formed to resolve institutional constraints serving as disincentives to cooperative involvement. In addition, this team will serve as a single point of contact for interested parties and represent their interests within NASA.

Emphasis will be placed on the expansion of currently funded activities for ground-based and spaceflight investigations to maximize the outputs from these opportunities. Initiatives requiring new hardware will be encouraged at a low level until funds can be made available. The expansion of current efforts is occurring as a result of focusing support for current spaceflight investigations by forming facility experiment teams to provide advice and identify future involvement. Emphasis has been placed on experiments involving the Materials Experiment Assembly and Mid-Deck experiments on the Space Shuttle. Our continuing research for more experimental opportunities on STS involves the Fluids Experiment System, the Solidification Experiments System, Acoustic Containerless Processing Module, and the Vacuum Crystal Growth System.

Goal

The program demonstrates the capabilities of the space environment for materials processing to the scientific and commercial user communities and provides opportunities for independently funded users to exploit the space environment for processing related to their own needs.

Objectives

The program has been structured to achieve the following specific objectives:

- Demonstrate the ability to achieve control over process variables at levels not achievable on Earth
 - Control thermal fields
 - Increase compositional uniformity in crystals
 - Lower defect concentrations in crystals
 - Align internal structure of metal alloy systems
 - Increase purity of optical glass systems
 - Increase stability range of new glass systems
 - Increase geometrical uniformity of glass microshells
 - Reduce self-deformation and dislocation density of growing crystals
 - Improve effectiveness of electrokinetic separation
 - Produce large-diameter monodispersed polymer latex spheres
- Develop and demonstrate the capabilities of containerless processing techniques to handle and measure the properties of molten, reactive materials on which experiments cannot be performed in Earth-based laboratories

- Viscosity
 - Equilibrium and dynamic vapor pressures
 - Enthalpy, emissivity, and temperature measurements
 - Phase equilibria studies
 - Enthalpy of solution and reaction
 - Processing of droplet arrays
- Demonstrate the nature of the vacuum achievable in space and its utility for extending the range of important experimental parameters in extra high vacuum science as well as its potential for achieving novel materials processing capabilities
 - Provide opportunities for independently funded scientific and commercial users to perform processing in the space environment

In addition to these activities, the program will also actively explore mission and hardware configurations which will reduce cost and provide sufficient power for experiments. In the period 1980-84, these definition studies will identify a series of phase options to provide such flight opportunities and then proceed with preliminary designs.

Low-Gravity Flight Projects

Low-gravity research is provided by several means to scientists to carry on experiments to verify ground-based predictions.

- a. Drop tube at Marshall Space Flight Center (MSFC); containerless solidification with approximately 2 seconds of weightlessness. There is also a new 300-foot tube that will be available in 1982.
- b. Drop tower at MSFC (approximately 4 seconds of free fall with a complete apparatus).
- c. KC-135 or F104 aircraft (up to 40 seconds of weightlessness in a Keplerian parabolic trajectory, available several times a year).
- d. SPAR (up to 5 minutes of weightlessness in coasting flight after power cut-off; samples recovered after parachute descent); more flights are planned.
- e. Materials Experiment Assembly (MEA); up to 5 days in orbit in the Space Shuttle. MEA is an autonomous package which will support three experiments with a minimum of integration effort

- f. Spacelab materials processing experiments (5 or more days of weightlessness on the Space Shuttle); hands-on experiments operated by Mission or Payload Specialists in a laboratory environment. First flight planned for Spacelab 3.
- g. Materials Experiments Carrier (MEC) will be an automatic, free-flying satellite to support a large number of experiments for 3 or more months.

Format

The key to the format for listing Materials Processing in Space program tasks in this catalog is as follows:

- a. Title
- b. Performing Organization
- c. Principal Investigator
- d. Co-Investigator(s)
- e. Contract Number and Total Cost
- f. Period of Performance
- g. Abstract of Objectives, Approach and Results
- h. Publications

Program Organization

The NASA Materials Processing in Space Program is administered by a Division Director and his staff in the Office of Space and Terrestrial Applications at NASA Headquarters, Washington, D.C. The Division Director is supported directly in the management of the program by the George C. Marshall Space Flight Center's Materials Processing in Space Project Office. That office depends upon the laboratories of the Marshall Space Flight Center, other NASA centers, other government agencies, universities, and industrial laboratories for technical support.

II. TASKS

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A. CRYSTAL GROWTH

Analysis of the Float Zone Process

Massachusetts Institute of Technology
Professor R. A. Brown
NSG-7645/Supplement 1

This research program is directed toward a fundamental understanding of the interaction of heat, mass, and momentum transfer in the floating zone method for growing single crystals from the melt.

Significant progress has been made on studies of the interaction between heat and mass transfer and melt/solid interface shape in melt growth processes, on the analysis of the fluid mechanics of the floating-zone process, and on the modelling of bouyancy-driven convection in crystal growth from the melt. In the first project, new finite element methods have been developed for calculating melt/solid interface shape in solidification processes and have been applied in a detailed study of heat and mass transfer in edge-defined film growth process.

The finite element methods have been extended to account for natural convection in the melt and its influence on melt/solid interface shape and solute segregation. Studies of prototypes of the vertical Bridgman system and the float zone processes are underway and results have been submitted for presentation. Asymptotic methods and finite element analysis have been used to study forced convection in the float zone system. Results of this study are being used to describe radial and axial segregation in systems operating in low-g conditions but in the absence of surface-tension-driven convection. The effects of Marangoni convection are being studied.

Publications

Ettourey, H. M. and Brown, R. A., "Finite Element Methods for Steady Solidification Problems," reviewed for publication in J. Comp. Phys.

Ettouney, H. M. and Brown, R. A., "Effect of Heat Transfer on Melt/Solid Interface Shape and Solute Segregation in Edge-Defined Film Growth: Finite Element Analysis," reviewed for publication in J. Crystal Growth.

Brown, R. A. and Chang, C. J., "The Effect of Natural Convection on the Shape of the Melt/Solid Interface," Bull Amer. Phys. Soc. 25 1100 (1980).

Chang, C. J. and Brown, R. A., "Finite Element Calculation of Natural Convection During Steady Solidification," reviewed for publication in J. Comp. Phys.

Chang, C. J. and Brown, R. A., "Radial Segregation Induced by Natural Convection and Melt/Solid Interface Curvature in Melt Crystal Growth," submitted for presentation at ACCG-5, San Diego, July 1981. Paper for J. Crystal Growth, in preparation.

Harriott, G. M. and Brown, R. A., "The Fluid Mechanics of a Differentially Rotated Captive Drop," J. Fluid Mech., in preparation.

Transient and Diffusion Analysis of HgCdTe

Semtec, Inc.
Dr. J. Creed Clayton
NAS8-33698 \$117K/year
December 19, 1980 - December 28, 1981

The goal of this effort is to analyze the directional solidification of the alloy system HgCdTe in order to obtain optimum processing conditions for crystal growth. Directional solidification of HgCdTe has been modeled and the results of this model have been applied to the initial and final solute segregation transients in order to estimate an effective diffusion constant. The one-dimensional, planar interface model incorporates aspects of the HgCdTe system that are not encountered in the classic doped semiconductor system. The model assumes diffusion-controlled solidification and treats the variation of interface temperature with composition, the variation of k with composition, and a growth rate determined by the thermal field. This treatment removes the assumptions that have made previous one-dimensional models inappropriate for the HgCdTe system.

Other tasks currently under study involve extending the model to two dimensions in order to investigate interface shape and stability. A method of deconvoluting the interface shape from experimental composition profiles and an assumed thermal field shows promising results. A potential interface shape instability due to sedimentation of the heavier HgTe solvent under a gravitational field is currently under study, and attempts to model this phenomenon are underway.

Publications

Clayton, J. C., "Transient and Diffusion Analysis of HgCdTe,"
NASA8-33698, Fifth Quarterly Report, December 20, 1980, 47 pgs.

Analysis of Directional Solidification Space Processing Experiments

Semtec, Inc.
Dr. J. Creed Clayton
NAS8-33746 \$96K/year
December 1979 - December 1981

This effort is to provide analysis support to MSFC personnel and the Principal Investigators selected for experiments to be performed in the Solidification Experiment System (SES). The SES and related Furnace Module (FM) comprise a complete system for crystal growth in space by a variety of techniques.

This study has concentrated on a set of four experiments to be processed by directional solidification. Semtec has created a thermal model of the furnace and experiment ampoules in order to assess the effects of various furnace temperatures, adiabatic zone lengths, and tickler heater widths on the resulting thermal profile in the ampoule. The thermal model is moderately simple and easy to run, thereby providing a "second opinion" to the results obtained from other much more complicated models.

In addition to further analysis of the final version of the furnace, an additional computer model is under development that makes use of assumed heat transfer coefficients for heat transfer between the furnace and sample. The purpose of this model is to link analytical models developed by MSFC personnel to numerical models used at Semtec and TRW. An additional use of this model will be to use experimentally measured heat transfer coefficients to predict the thermal field in those ampoules that cannot be adequately instrumented with thermocouples.

Publications

Clayton, J. C., "Analysis of Directional Solidification Space Processing Experiments," Annual Report, December 11, 1979-December 11, 1980, 107 pgs.

Solutal Convection During Directional Solidification

National Bureau of Standards
S. R. Coriell
R. J. Schaefer
H-27954B \$160K/year
April 1977 - continuing task

The objective of this task is to calculate and measure effects of convection caused by simultaneous temperature and concentration gradients on directional solidification, including determination of segregation effects in experiments done on Earth and estimation of the effect of micro-gravity and magnetic fields in avoiding such convection.

Theoretical methods are being developed to predict convective and interfacial instabilities during unidirectional solidification of binary alloys in the vertical direction. Linear hydrodynamic stability theory incorporating solidification boundary conditions has been formulated and has established some criteria for the onset of nonplanar solidification and nonuniform incorporation of solute in the resulting solid, including effects of magnetic fields. This research will delineate the conditions under which gravity-driven convection will occur during constant velocity unidirectional solidification.

Experimental studies of instabilities are being carried out in succinonitrile doped with small additions of ethanol. The concentration and unidirectional solidification velocities have been selected to be close to the values for which the theory predicts transitions from stability to instability. Convection is detected by observations of small neutrally buoyant particles in the liquid, and the behavior of the doped material is compared to that of ultra-high purity material in which any convective flow must be attributed to thermal gradients or solidification shrinkage.

Publications

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 78-1483, J. R. Manning and L. L. Parker, Eds., 1978, pp. 15-68.

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 79-1767, J. R. Manning, Ed., 1979, pp. 25-81.

Coriell, S. R., Cordes, M. R., et al., "Convective and Interfacial Instabilities During Unidirectional Solidification of a Binary Alloy," J. Cryst. Growth, 49, May 1980, pp. 13-28.

Sekerka, R. F. and Coriell, S. R., "Influence of the Space Environment on Some Materials Processing Phenomena," Proceedings of Third European Symposium on Materials Sciences in Space, Grenoble, France, April 1979, ESA SP-142, pp. 55-65.

Coriell, S. R., Cordes, M. R. , et al., "Effect of Gravity on Coupled Convective and Interfacial Instabilities During Directional Solidification," Proceedings of Twenty-third COSPAR Plenary Meeting, Budapest, Hungary, 1980, pp. 5-11.

Boettinger, W. J., Coriell, S. R., et al., "Solutal Convection Induced Macrosegregation and the Dendrite to Composite Transition in Off-Eutectic Alloys," Met. Trans. 12A, 321-327 (February 1981).

Semiconductor Material Growth in Low-G Environment

Langley Research Center
Dr. R. K. Crouch
Dr. A. L. Fripp
In-Center Total Cost: \$260K
February 1978 - February 1983

The principal purpose of this experiment is to utilize the microgravity environment of space to investigate the effect of convection on the homogeneity and perfection of compound semiconductor crystals. In a gravity field, the specific material $Pb_{1-x}Sn_xTe$ being investigated has unstable solutal gradients or unstable thermal gradients depending on growth orientation.

Three different growth processes will be considered: (1) a vapor phase sublimation for seeded growth, (2) a modified Bridgman growth in which polycrystalline aggregate is necked down to encourage growth of a single crystal, and (3) a modified Bridgman melt back and regrowth.

Research in preparation for the space flight consists of both theoretical and experimental efforts. Numerical analysis of the mass and heat transfer will predict the furnace profile needed to obtain a planar isotherm at the melt-solid interface. Crystals grown on Earth will be used for comparison with those grown in space.

Publications

Crouch, R. K., et al., "Growth of $Pb_{1-x}Sn_xTe$: Earth Options and Space Opportunities," 157th Electrochemical Society Meeting, St. Louis, Mo., May 1980.

Crouch, R. K., et al., "High Gradient Furnace Studies Using Modular Design," Gordon Conference on Crystal Growth, Santa Barbara, CA, January 1979.

Crouch, R. K., Fripp, A. L., et al., "Experiment Requirements and Implementation Plan (ERIP) for Semiconductor Growth in Low-g Environment, Experiment No. MPS 77F087," NASA TM-81943, February 1981.

Clark, I. O., Fripp, A. L., et al., "Diffusion Coefficients for Liquid $Pb_{1-x}Sn_xTe$," 157th Electrochemical Society Meeting, St. Louis, MO, May 1980.

Clark, I. O., Fripp, A. L., et al., "Solutal Diffusion Coefficient for Liquid $PbTe-Sn-Te$," to be submitted to J. Electrochem. Soc.

Frapp, A. L. and Crouch, R. K., "Compositional Control Required in Alloy Semiconductors Used in High Performance Infrared Detector Arrays," Infrared Phys. 19, 701 (1979).

Frapp, A. L., et al., "Epitaxial $Pb_{1-x}Sn_xTe$ on Ba_2 Substrate," J. Electrochem. Soc. 124, 104C (1977).

Debnam, W. J., Frapp, A. L., and Crouch, R. K., "Reusable Clamp That Can Undergo Thermal Cycling for Fused Quartz to Metal Attachment," disclosure of invention, November 20, 1980.

Debnam, W. J. and Clark, I. O., "A Technique for Precision Sealing Fused Quartz Ampoules Without Loss of Vacuum Pumping Speed and Without Loss of Wall Strength," disclosure of invention, October 1980.

Growth of Solid Solution Crystals

Athens State College

Dr. L. R. Holland

Dr. A. F. Witt, MIT

Dr. D. D. Schenk, BMD-ATC

In-House Total Cost: \$540K

October 1977 - October 1982

The major objective of this program is to determine the conditions under which single crystals of solid solutions can be grown from the melt in a Bridgman configuration with a high degree of chemical homogeneity. The central aim of this program is to assess the role of gravity in the growth process and to explore the possible advantages for growth in the absence of gravity. Since HgTe rejected at the growth interface is more dense than the bulk melt, the system may be stable against thermosolutal convection, but the negative thermal expansion of the melt may be sufficient to upset this stability in a high thermal gradient. The pressure of a dense component that lowers the melting point of the solid also gives rise to a gravity-dependent interface shape instability. Finally, the interaction between unavoidable radial thermal gradients in the melt and the diffusion region may produce radial homogeneities. The primary focus to investigate the role of gravity in these processes.

The problems of purity and containment in quartz ampoules have been resolved. The necessary purity and the resulting absence of chemical attack on the quartz was achieved by obtaining ultrapure starting material and loading by distillation. The structural integrity of the ampoules at the high vapor pressures associated with growth of this system was demonstrated. Crystals were grown by the Bridgman method and analyzed by the energy dispersive X-ray technique (Kevex). Composition was determined longitudinally and radially. These compositional profiles are being analyzed by one-dimensional and two-dimensional models. In addition to the basic studies, thermal profiles were determined to obtain the optimum growth environment for the HgCdTe material.

Publications

Davidson, M. C. and Holland, L. R., "Narrow Zone Heating by a New Radiation Focusing Technique - Toroidal Ellipsoid Furnace," Rev. Sci. Instrum. 49, 1156-1159 (August 1978).

Holland, L. R., "A Thermal Transmission Function for Fused Silica Ampoules," J. Cryst. Growth 49, 246 (1980).

Marangoni Effect in Crystal Processing

Arthur D. Little
Dr. Arthur A. Fowle
Dr. A. F. Witt, MIT
NAS8-32946, Total Cost: \$552K
March 1978 - December 1980

The Spacelab experiment consists of measuring the freezing interface morphology and the velocity and temperature fields on the surface of a molten zone in a cylindrical sample of gallium-doped germanium in a crystal growing configuration. The nominal sample diameter is 0.7 cm, and the pulling rate is variable. Postflight characterization of the space-grown crystals will be made, and the results of the measured experimental variables will be compared with theory.

The experiment will be carried out under conditions in which molecular diffusion-controlled processes can dominate the behavior of the melt because gravity-induced convection is made negligible in the microgravity environment of Spacelab. This processing condition is desirable because the scientific understanding and control of it would produce an immediate promise for producing better semiconductor, crystal products. However, flows driven by surface tension gradient (Marangoni effect) are predicted to upset the diffusion-controlled process ideal. A major purpose of the experiment, and the theory in support of it, is to examine the nature and importance of the Marangoni effect.

Publications

Fowle, A. A., "Float Zone Experiments System (FZES) for Spacelab," 17th Aerospace Sciences Meeting, New Orleans, LA, January 1979. AIAA Paper 79-0371.

Fowle, A. A., et al., "Experiments on the Stability of Conical and Cylindrical Liquid Columns at Low Bond Numbers," Third European Symposium on Material Sciences in Space, Grenoble, France, April 1979, ESA SP-142, pp. 317-325.

Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology

Professor H. C. Gatos

Dr. Jacek Lagowski

NSG 7331 \$350K/year

April 1, 1977 - continuing task

The experimental and theoretical efforts are aimed at the establishment of relationships among crystal growth parameters, materials properties, electronic properties and device applications of GaAs. Toward this goal steps have been undertaken for the development of new approaches to the preparation and characterization of GaAs. This extensive ground-based program constitutes a necessary step toward insuring successful processing of GaAs under zero gravity conditions. Due to its unique scope combining crystal growth characterization and device-related properties and phenomena, this program bears directly upon exploitation of the potential of GaAs in device applications.

The research task includes the detailed study of the mechanisms of GaAs crystal growth from the melt and from solution and of the development of techniques for the characterization of materials and electronic properties on a microscale, e.g., composition, carrier concentration, mobility, diffusion length, and lifetime. Relationships between electronic properties and device performance are a part of the research task.

Publications

Jastrzebski, L., Lagowski, J., et al., "Liquid-Phase Electroepitaxy: Growth Kinetics," J. Appl. Phys. 49, 5909 (1978).

Jastrzebski, L., Lagowski, J., et al., "Determination of Carrier Concentration and Compensation Microprofiles in GaAs," J. Appl. Phys. 51, 2301 (1980).

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Kamieniecki, E., Lagowski, J., et al., "Wavelength Modulated Photo-Capacitance Spectroscopy," J. Appl. Phys. 51, 1863 (1980).

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III-V Semiconductor Solid Solution Single Crystal Growth

Rockwell International

Dr. E. R. Gertner

Dr. M. D. Lind

JPL 955354 Total Cost: \$146K

April 6, 1979 - December 6, 1980

The overall program task is to improve the quality of semiconductor substrate material used in epitaxial growth processes, since the quality of the epitaxial deposit is often limited by the quality of the substrate. To achieve the desired material improvement, the floating zone technique used in conjunction with a low-gravity environment will be used. This combination of technique and environment is capable of producing large, uniformly doped and compositionally homogeneous single crystals. The III-V solid solutions were selected for the initial growth experiments. Strain-free, homogeneous single crystals of III-V solid solutions have not been successfully synthesized due to the nature of their phase diagrams where the liquids and solids are of different composition. The $\text{Ga}_{1-x}\text{In}_x\text{Sb}$ was chosen initially because it has the lowest melting point and vapor pressure of the III-V solid solutions. The research objective was to generate a compositionally homogeneous seed for future float zone experiments. However, technical difficulties with the original approach and recent advances in the vapor growth of III-V solid solution led to a reassessment of the initial approach and a redirection of the program to the single crystal growth of CdTe, a II-IV compound.

The research task will involve attempts to float zone small-diameter CdTe crystals because density, surface tension, and thermal conductivity considerations for CdTe preclude the use of float zoning for large crystals in normal gravity. Materials synthesized will be analyzed by optical microscopy, X-ray diffraction and topography, infrared spectroscopy, and electrical measurements.

Characterization of Semiconductor Materials

Universities Space Research Association

Dr. D. C. Gillies

In-House

October 1980 - October 1981

The objective of this experiment is to develop techniques for characterizing high-quality, solid-solution, alloy-type semiconductors for use as infrared detectors or as IR transparent substrates. IR detectors can be grown by LPE or other techniques. Emphasis has been given to $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$ because of its importance as a detector material and because it is generally accepted as one of the most difficult materials of this type to grow on Earth. One of the major goals is to achieve a very low intrinsic carrier concentration in order to extend the bandwidth from the existing 2 GHz to approximately 5 GHz to satisfy a Department of Defense (DOD) requirement.

Chemical analysis data for melt growth experiments are necessary to verify computed models simulating crystal growth. One-dimensional models enable diffusion coefficients to be obtained from transient data and translation rate experiments. It is anticipated that some assessment of radial diffusion and fluid flow along the interface will be obtained from 2-dimensional analysis. In addition, crystal growth will be interrupted in an attempt to quench in the interface. High spatial resolution results are needed to locate this interface. An X-ray analysis technique employing an energy dispersive system on a scanning electron microscope has been devised for the determination of x in $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$. The options exist for using a range of standards of different compositions and analyzing different spectral lines (up to 100 points per day can be analyzed). Additional tasks being performed include the determination of x on a macro scale by measuring the infrared cut-off wavelength, metallographic studies of etch pits, inclusions and grain boundaries and crystal perfection and orientation studies of x-ray back reflection Laue techniques.

Solution Growth of Crystals in Zero-Gravity

Alabama A&M University

Dr. R. B. Lal

Dr. R. L. Kroes, MSFC

NAS8-32945 Total Cost: \$492K

June 28, 1978 - June 28, 1983

In a low-g environment, buoyancy-driven convection effects in solution crystal growth are greatly reduced and, thus, one can study diffusion mass transport which in 1-g is masked by convective phenomena. Also, triglycine sulfate (TGS) crystals have technological importance for infrared detectors. The objectives of the experiment are (1) to grow TGS crystals from aqueous solution in low-gravity, (2) to investigate mass transport and heat flow in a diffusion-controlled growth system, and (3) to evaluate the feasibility, possible advantages and technical potential of producing solution growth crystals in space.

Single crystals of TGS have been grown using conventional low-temperature solution growth method, and the growth process has been extensively characterized. Also, a unique technique of growing solution growth crystals by extracting heat at a programmed rate from the crystal through a semi-insulating sting has been developed. TGS crystals will be grown by this technique during the Spacelab 3 mission. Data on heat and mass transport in a diffusion-controlled system will be obtained using a laser holography technique. Analytical studies are under way to estimate growth rates in low-g conditions.

Publications

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Aligned Magnetic Composites

Grumman Aerospace Corporation
Dr. D. J. Larson, Jr.
NAS8-32948 Total Cost: \$652K
July 1978 - July 1983

The objectives of this program are to contribute to understanding the role of convection on plane front solidification of eutectic and peritectic composites and the relationships between morphology and magnetic properties. In addition, assessment will be made of the commercial potential for processing binary composites in low-g.

The aim of this program is to evaluate the impact of convection (thermal and/or solutal) or coupled convective/diffusive transport on the plane front solidification of contained binary magnetic composites. Eutectic, off-eutectic, and peritectic solidification are under investigation. The low-g orbital environment will be utilized to study diffusion-controlled solidification for experimental regimes that would be described as convective/diffusive regimes terrestrially. In addition, the relationships between solidification processing parameters, solidification microstructure, microchemistry, and magnetic properties are being studied.

Publications

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Advanced Methods for Preparation and Characterization of
Infrared-Detector Materials

McDonnell Douglas Research Laboratories

Dr. S. L. Lehoczky

Dr. F. R. Szofran

Dr. B. G. Martin

NAS8-33107 \$80K/year

December 1978 - December 1981

The objectives of this - search program are to quantitatively establish the characteristics of $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ as grown on Earth (1-g) as a basis for subsequent evaluation of the material processed in space and to develop experimental, theoretical, and analytical methods required for such evaluation.

A series of differential thermal-arrest (DTA) measurements were performed for $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ alloy compositions $x = 0, 0.1, 0.2, 0.3, 0.4, 0.6, 0.7, 0.8, 0.9, 1.0$. The solidus and liquidus temperatures deduced from the DTA data were used to establish the pseudobinary HgTe-CdTe constitutional phase diagram and the x and interface temperature dependencies of the Cd segregation coefficient, k . Iterative phase-equilibria calculations, based on a regular associated solution (RAS) theory, were performed to establish the solution parameters required to calculate the phase boundaries for the Hg-Cd-Te alloy system. Further DTA measurements and theoretical analysis are being performed to determine phase equilibria parameters for selected regions of the ternary Hg-Cd-Te phase diagram.

Theoretical models and computer programs specific to $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ were developed for calculations of charge-carrier concentrations, Hall coefficient, Fermi energy, and conduction-electron mobility as functions of x , temperature, an ionized-defect and neutral-defect concentrations. A comparison of calculated results with available experimental data indicated that longitudinal optical-phonon and charged and neutral defect scattering are the dominant mobility limiting mechanisms.

Publications

Szofran, F. R. and Lehoczky, S. L., "The HgTe-CdTe Pseudobinary Phase Diagram," 22nd Annual Electronic Materials Conference, Ithaca, New York, June 24-27, 1980.

Lehoczky, S. L., Szofran, F. R., et al., "Advanced Methods for Preparation and Characterization of Infrared Detector Materials," NAS8-33107, Final Report (Period December 1978-July 1980).

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Szofran, F. R. and Lehoczký, S. L., "Radial Homogeneity of Directionally Solidified $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$," Materials Research Society Symposium - Materials Processing in the Reduced Gravity Environment of Space, Boston, MA, November 1981 (to be submitted).

Epitaxial Growth of Single Crystal Films

Rockwell International

Dr. M. David Lind

Dr. R. L. Kroes, MSFC

NAS8-31733 Total Cost: \$138K

October 14, 1975 - May 31, 1980

The objective is to grow epitaxial films of gallium arsenide by liquid phase epitaxy (LPE) in low gravity and to compare them with films grown in normal gravity. To investigate crystal growth in low gravity, an experiment was designed and performed on a Space Processing Applications Rocket (SPAR) flight. GaAs LPE appeared to be an appropriate process for investigation in such a flight because it can be completed within the few minutes of low gravity provided by the flight and because of broad interest in this process and material.

A general purpose LPE processor suitable for either SPAR or Space Transportation System flights was designed and built. It incorporates a tubular resistance heated furnace, a system to provide a flowing H_2 atmosphere inside the furnace, a pneumatically operated slider, and an electronic process controller. For the SPAR flight the process was started before the launch, and only the final step, in which the epitaxial film is grown, was performed during the flight. For a planned growth temperature of $720^\circ C$, the solution (As in excess Ga) was saturated at $730^\circ C$. Ninety minutes before the launch the furnace temperature was raised to $780^\circ C$ and then, beginning at the launch, was lowered at a rate that allowed the growth temperature to be reached within the first four minutes of the flight. By then, the near-zero gravity condition was well established, and the fluid motion induced by the launch was damped out. Growth was initiated at this time by sliding 2 GaAs substrate wafers into contact with the solution. It was terminated after 1 minute, retracting the substrates.

The experiment achieved its objectives. Epitaxial films of reasonably good quality and very nearly the thickness ($\sim 1 \mu m$) predicted for convection-free, diffusion-limited growth were produced. The films have been examined by conventional analytical techniques, including electrical and photoluminescence measurements and x-ray diffraction topography. The results are satisfactory for an initial space flight experiment and provide ample justification for further experimentation with LPE processes in the absence of gravity.

Publications

Lind, M. D., Kroes, R. L., et al., "Liquid Phase Epitaxy (LPE) of Gallium Arsenide in Low Gravity," 157th Electrochemical Society Meeting, St. Louis, MO, May 1980.

Lind, M. D., "Epitaxial Growth of Single Crystal Films," SPAR III Final Report, NASA TM-78137, January 1978.

Lind, M. D., "Crystal Growth from Solutions in Low Gravity," 15th Aerospace Sciences Meeting, Los Angeles, CA, January 24-26, 1977, AIAA Paper 77-197.

Lind, M. D., "Crystal Growth from Solutions in Low Gravity," AIAA Journal 16, 458-462 (May 1978).

Analytical Approach to Modeling of Heat Flow in Bridgman-Type Crystal Growth

Marshall Space Flight Center

Dr. R. J. Naumann

Ms. Ernestine Cothran

In-House

October 1980 - May 1981

The purpose of this program task is to develop an analytical approach to the modeling of heat flow in Bridgman-type crystal growth. Approximate analytical techniques have been developed for obtaining the temperature field and isotherms in crystals being grown from the melt by the vertical Bridgman technique. One-dimensional models are useful for low Biot numbers and have been used to estimate the thermal profiles, determine the position and motion of the growth interface, and assess the axial thermal gradients in the sample as functions of furnace and sample parameters, sample insertion length, and sample motion. This analysis is particularly valuable for establishing control conditions for meeting processing requirements, for performing sensitivity analyses, and for performing engineering trade studies such as optimizing the length of the booster heater zone for enhancing axial thermal gradients in the sample.

A two-dimensional analysis has also been developed which can accommodate different thermal properties of the sample in the melt and solid phases and can locate the position and determine the shape of the solidification interface in a 3-zone furnace which includes an insulated or adiabatic zone. Finally, limiting cases involving very large Biot numbers were considered. It has been shown that the maximum axial gradient in a long cylindrical samples that can be obtained by the Bridgman technique is approximately $2/3$ times the difference between the hot and cold end temperatures divided by the sample radius. Effects of sample motion and length of the adiabatic zone were also examined for this limiting case.

Publications

Naumann, R. J., "A Novel Technique for Obtaining Fourier Bessel Coefficients in Cylindrical Heat Flow Problems," Seminar to Math Department, University of Alabama, Huntsville, 1980.

Naumann, R. J., "Optimization of Axial and Radial Thermal Gradients in Vertical Bridgman Growth," Seminar to Materials Science Department, MIT, 1981.

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Ultravacuum Vapor Epitaxial Growth of Silicon

General Electric Company

Dr. C. A. Neugebauer

NAS8-33121 Total Cost: \$105K

August 30, 1978 - March 31, 1980

The objective of this program is to develop an ultra-high vacuum deposition process for the growth of polycrystals of silicon onto a polycrystalline oriented metal substrate. An important objective is to establish vacuum and vacuum purity requirements for the process and possible rationale for extension of these experiments using the greatly improved vacuum conditions and pumping speeds available in a Space Vacuum Research Facility. In addition, experiment requirements for possible extension of the vacuum deposition work in a Space Vacuum Research Facility are to be documented. Support of the system definition design work for such a facility is to be provided.

Silicon films 5 to 10 microns thick were deposited onto 25 to 50 micron tungsten foils from a resistively heated silicon source. Vacuum levels in the low 10^{-8} Pa range and substrate temperatures in the 450°C to 600°C range were employed. X-ray diffractometry, scanning and transmission electron microscopy, and metallographic examination of cross-sectioned and etched films were among the methods used to examine the deposited films.

It was concluded that oriented deposits can be obtained at substrate temperatures below 600°C. Two types of Si crystallites were distinguished: subgrains of ~1 micron or less diameter which were columnar and only slightly misaligned with respect to each other, and large parent grains subtending many subgrains and coinciding with the tungsten grains in the substrate. Increasing the gas pressure leads to more random deposits, indicating lower pressure might lead to more ordered growth.

Fluid Dynamics of Crystallization from Vapors

University of Utah - Salt Lake City

Dr. F. Rosenberger

NSG-1534 Total Cost: \$271K

June 1, 1978 - May 31, 1981

This program is aimed at obtaining fundamental insight into the complex physiochemical fluid dynamics of closed ampoule vapor crystal growth processes to the extent that a desired set of crystal growth conditions can be designed in advance. A more directly applied part of the program is concerned with the synthesis of ultrapure mercuric iodide and the vapor composition (stoichiometry) required for the growth of mercuric iodide high resolution radiation detector crystals.

Numerical modeling of vapor transport in vertical ampoules has shown that diffusion fluxes, in viscous interaction with the wall, establish density gradients normal to the main transport direction. These density gradients act convectively destabilizing even in ampoule orientations which, classically, were considered convection-free (e.g., "heating from top"). Also, it has been demonstrated that the convection behavior in crystal growth ampoules cannot be extrapolated from known solutions to fluid dynamically "similar" monocomponent (pure) systems. The net transport across the vapor space causes drastic changes as compared to convection patterns in cylinders with impermeable end faces. It has been demonstrated experimentally that thermal diffusion in ampoules acts convectively more destabilizing than in laterally unbound (Benard) geometries. Modeling of vapor transport across a horizontal cavity has shown that at lower transport rates earlier, simplifying treatments (Klosse-Ullersma, KU) give reasonable results because of fortuitous cancellation of errors. Disagreements between experimental transport rates obtained by other authors and the KU-model have been traced to solutal convection contributions not accounted for.

Publications

Rosenberger, F., "Fluid Dynamics in Crystal Growth from Vapors," J. Physchem. Hydrodynamics, 1, (1980).

Markham, B. L. and Rosenberger, F., "Velocity and Concentration Distribution in Stefan Diffusion Tubes," Chem. Engr. Commun. 5, 287 (1980).

Markham, B. L., Greenwall, D. W., et al., "Numerical Modeling of Physical Vapor Transport in Vertical Ampoules," J. Crystal Growth 51, 426 (1981).

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Jhaveri, B. S. and Rosenberger, F., "On Singular Boundary Conditions in Diffusive-Convective Mass Transfer Across a Cavity," in preparation.

HgI₂ Crystal Growth for Nuclear Detectors

EG&G, Inc.
W. F. Schnepple
Dr. L. Van den Berg
H-34318B Total Cost: \$646K
April 28, 1978 - April 28, 1983

The objectives of this program are to obtain a benchmark quality sample grown at low-g conditions and to study vapor growth phenomena under space conditions.

Ground-based crystals show a defect structure which impairs their performance as nuclear radiation detectors. These defects may be caused by the gravitational force acting on the crystal in its weakened state at the elevated growth temperature and by irregular convection patterns in the vapor during growth.

The program will be supported by ground-based research aimed at a more detailed understanding and description of the problems associated with the crystal growth process. Mechanical strength measurements have been performed (uniaxial compression tests) which show that the crystals exhibit slip parallel to the c-planes at stresses as low as 10 psi. Preliminary calculations using a simple linearized model indicate the oscillating instabilities in the convection part of the vapor transport system are unlikely, even at 1 g, provided that the utmost care is taken in the preparation of the crystal growth source material.

Publications

Schieber, M., Schnepple, W. F., et al., "Vapor Growth of HgI₂ by Periodic Source or Crystal Temperature Oscillations," J. Cryst. Growth, 33, 1979, p. 125.

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Twanczyk, J. S., Dabrowski, A. J., et al., "A Study of Low-Noise Preamplifier Systems for Use with Room Temperature Mercuric Iodide (HgI₂) X-Ray Detectors," IEEE Trans. Nuc. Sci., NS-28, 579 (1981).

Crystal Growth in a Spaceflight Environment

Jet Propulsion Laboratory
Dr. P. J. Shlichta
NAS7-100 \$118K/year
October 1979 - October 1980

Experiments being developed for early Spacelab missions are directed at (1) determining the effects of residual and transient accelerations on the growth process and on resultant crystal perfection, (2) determining the effect of low-level forced convection, and (3) developing a better understanding of the crystallization process by measuring growth rates versus thermal and concentration gradients in the absence of convection.

Crystals will be grown from solutions at programmed cooling rates in controlled thermal geometries. Cameras will record time-lapse sequences of processed images of the experiment, e.g., by holography, schlieren, shadowgraphs, monochromatic absorption, interferometry, and/or reflected light. Analysis of these images will provide maps of the growth rate of the crystal and the temperature, concentration, and motion of the surrounding solution. These data will be compared with the distribution of defects and impurities in the crystal and with the acceleration history of the space flight. Results will provide guidelines for optimum experimental parameters for future Spacelab crystal growth experiments.

Publications

Chen, P. S., Shlichta, P. J., et al., "Convection Phenomena during the Growth of Sodium Chlorate Crystals from Solution," J. Cryst., Growth, Vol. 47, 1979, pp. 43-60.

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31 March 1980).

Direct Observation of Interf. & Stability

Stanford University
Professor W. A. Tiller
Professor R. S. Fiegelson
Dr. Dennis Elwell
NAS8-33110 Total Cost: \$265K
December 1, 1978 - January 31, 1982

This task will use a direct observation method to facilitate the careful test of interface stability criteria in a convective environment. This study will form the basis for future experiments under nonconvective conditions. The objective is a careful test of theory with experiment on a model system with all the significant material parameters being measured for this system. Such a careful test has not been performed heretofore.

The relevant parameters will be measured, including: (1) diffusion coefficients of the solutes in the liquid, (2) phase diagram and effective distribution coefficients for the solute, (3) liquidus slopes for the chosen solutes, (4) thermal conductivities and diffusivities for both liquid and solid, and (5) the solid-liquid interfacial energy.

Publications

Tiller, W. A. and Ahn, K. S., "Interface Field Effects on Solute Redistribution During Crystallization," J. Cryst. Growth 49, 483-501 (1980).

Ahn, K. S., Carranza, R. A., et al., "Crystal Growth of Cesium Cadmium Chloride CsCdCl_3 ," J. Cryst. Growth 50, 775-778 (1980).

Float Zone Experiments in Space

Ames Laboratory, Iowa State University
Dr. J. D. Verhoeven
H-343288 \$82K/year
October 1981 - October 1982

The objectives of the program were: (1) to determine if surface tension-driven convection in a float zone can be controlled or eliminated by means of surface films and (2) to investigate solute distribution and measure liquid diffusion coefficients in floating zones. If surface tension-driven convection can be effectively controlled or eliminated, it should be possible to obtain homogeneous solute distributions in space, which is not possible on Earth. Also, it should be possible to obtain accurate liquid diffusion coefficients for systems too reactive to be contained in capillary tubes.

Ground-based experimental work involves building an experimental float zone apparatus which will allow high vacuum capability plus control and measurement of temperature gradient, growth rate, and oxygen potential. Oxide layer thicknesses will be evaluated by depth profiling plus Auger analysis. For program task No. 1, experiments will consist of measuring the effect of oxide layer thicknesses upon interface shapes, and the radial and longitudinal composition profiles. For program task No. 2, solute profiles will be evaluated in the initial transient zones and the quenched zones.

Defect Chemistry and Characterization of (Hg, Cd) Te

Honeywell

Dr. H. R. Vydyanath

NAS8-33245 Total Cost: \$246K

December 18, 1978 - December 17, 1981

($\text{Hg}_{1-x}\text{Cd}_x$)Te is a variable bandgap alloy semiconductor extensively used for infrared applications. Detector performance close to theoretical limits imposed by infrared imaging systems requires extreme control of compositional uniformity, carrier concentration, and carrier lifetime. The program deals with the study of the nature and concentration of the lattice defects incorporated into ($\text{Hg}_{1-x}\text{Cd}_x$)Te alloys as a function of the physiochemical conditions of preparation; namely, the temperature, the partial pressures of the constituent elements, and/or the concentration or the activity of the dopant being incorporated.

Undoped, donor-doped, and acceptor-doped ($\text{Hg}_{1-x}\text{Cd}_x$)Te samples are annealed at various temperatures in suitable Hg atmospheres. The samples are quenched to room temperature from the high temperatures. Hall effect and resistivity measurements are carried out at 77 K to determine the carrier concentrations and mobilities. The variation of the carrier concentrations as a function of the partial pressure of Hg and/or the dopant concentration is used to arrive at defect models for the doped and the undoped crystals,

At the end of the 24-month period of the program, significant accomplishments have been made toward understanding the nature of lattice defects and the mode of incorporation of different dopants. For the first time in literature, the defect structures of undoped $\text{Hg}_{0.2}\text{Te}(\text{S})$, undoped, copper-doped and indium-doped $\text{Hg}_{0.2}\text{Te}(\text{S})$ have been established. The native acceptor defects have been found to be doubly ionized in both $\text{Hg}_{0.6}\text{Cd}_{0.4}\text{Te}(\text{S})$ and $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}(\text{S})$. Native donor defects are found to be negligible in concentration in these alloys, and the origin of p-type to n-type conversion has been shown to be due to residual foreign donors and not due to native donor defects. Thermodynamic constants for the incorporation of the native acceptor defect has been established in both $\text{Hg}_{0.6}\text{Cd}_{0.4}\text{Te}(\text{S})$ and $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}(\text{S})$.

Publications

Vydyanath, H. R., Nelson, D. A., et al., "($\text{Hg}_{1-x}\text{Cd}_x$)Te Materials Studies," Proceedings of the IRIS Detector Specialty Group Meeting, Minneapolis, MN, June 15-17, 1979, p. 29.

Vydyanath, H. R., Nelson, D. A., and Lancaster, R. A., "Defect Chemistry and Characterization of $(\text{Hg}_{1-x}\text{Cd}_x)\text{Te}$," J. of Electrochem. Soc. **126**, 371C (1979).

Vydyanath, H. R., "Defect Studies in $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$," presented at the Conference on the "Crystal Growth and Characterization of II-VI Compounds", University of Lancaster, U.K., April 14-16, 1980.

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Vydyanath, H. R., "Lattice Defects in $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ Alloys, II. Defect Structure of Indium Doped $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$, Accepted for publication in Journal of Electrochemical Society, 1981.

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Vydyanath, H. R. and Kroger, S. A., "Doping Behavior of Iodine in Mercury $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$," to be published in Journal of Electrochemical Society, January 1982.

Vapor Growth of Alloy-Type Semiconductor Crystals

Rensselaer Polytechnic Institute

Dr. Heribert Wiedemeier

NAS8-32936 Total Cost: \$460K

March 1978 - March 1983

The objectives of this program are to investigate through systematic ground-based studies the effects of gravity-driven convection on the growth of single crystals of alloy-type semiconductors, to define optimum conditions for the growth of these materials in a microgravity environment, and to perform crystal growth studies in space. For this purpose, the systems $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$ and CuInS_2 have been selected.

The ground-based research is concerned with the quantitative investigation of the chemical transport properties of the preceding materials in evacuated, closed ampoules of fused silica employing elemental iodine or metal-iodides as transport agents. The mass transport rates and crystal morphology of these systems are investigated as a function of total pressure for different temperature gradients and under horizontal and vertical stabilizing conditions. The inherent partial pressure and density gradients of the system and the presence of gravitational forces on Earth cause convective interferences with the transport and condensation process. Present results reveal that the surface morphology and chemical homogeneity of $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$ crystals obtained under vertical stabilizing conditions are improved relative to crystals grown under horizontal conditions. The crystal quality of CuInS_2 shows similar improvements for the horizontal ampoule configuration with decreasing pressure (decreasing convective interference) of the system. The combined results of ground-based studies will lead to the definition of optimum growth conditions for the actual space experiments.

Publications

Wiedemeier, H., Chandra, D., et al., "Diffusive and Convective Vapor Transport in the GeSe-GeI_4 System," J. Cryst. Growth, in press.

Wiedemeier, H., Gaur, U., et al., "Analysis of the Heat Capacities of Group IV Chalcogenides Using Debye Temperatures," Journal Thermal Analysis, in press.

Wiedemeier, H. and Chandra, D., "Chemical Vapor Transport and Thermal Behavior of the GeSe-GeI_4 System for Different Inclinations with Respect to the Gravity Vector, Comparison with Theoretical and Microgravity Data," J. Cryst. Growth **51**, 345-361 (February 1981).

Fluid Dynamics and Thermodynamics of Vapor Phase Crystal Growth

Rensselaer Polytechnic Institute
Dr. Heribert Wiedemeier
NAS8-33562 Total Cost: \$254K
January 1, 1980 - December 31, 1982

The primary objective of this program is to provide basic mass transport and crystal growth data which, combined with a thorough knowledge of the thermodynamics, will improve the fluid dynamic characterization of vapor transport systems.

The program is concerned with the investigation of the effect of systematic variations (1) of the relative importance of buoyancy-driven convection and (2) of diffusion and viscosity conditions on mass transport and crystal growth. These investigations will be performed in evacuated, closed ampoules for selected temperature gradients and partial pressures of the transport species. The specific experimental tasks include mass transport rate studies as a function of ampoule orientation, geometry, and the effects of inert gas additions. These will be correlated with crystal growth rate and morphology studies. In addition, the analysis of the vapor phase is an essential aspect of this project. The experimental efforts are supported by theoretical studies, including the thermodynamic and fluid dynamic characterization of the gas phase and the estimation of mass transport rates for different diffusive and diffusive-convective conditions. The materials investigated under this program include selected group IV elements and IV-VI compounds and halogens as transport agents. This choice is based on increasing complexity, known thermochemical and structural properties of these materials, and the existence of microgravity results for some of these systems investigated in previous Skylab and ASTP experiments.

Publications

Csillag, F. J. and Wiedemeier, H., "Equilibrium Sublimation and Thermodynamic Properties of SnS ," Thermochim. Acta. **34**, 257 (1979).

Csillag, F. J., Gaur, U., et al., "Heat Capacity Measurements on Sn_2S_3 and the Thermodynamic Functions of the Tin Sulfides," Thermochim. Acta **35**, 187 (1980).

Csillag, F. J. and Wiedemeier, H., "The Decomposition and Thermodynamic Properties of Sn_2S_3 ," Z. Anorg. Allg. Chem. **469**, 197 (1980).

Csillag, F. J. and Wiedemeier, H., "The Decomposition and Thermodynamic Properties of SnS_2 ," High Temp. Science, in press.

Von Schnering, H. G. and Wiedemeier, H., "The High Temperature Structure of β -SnS and the β 16-to-B 33 Type-Transition Path," Z. Kristallog., in press.

Pultz, G., Gaur, U., et al., "Heat Capacity Measurements of SnSe and SnSe₂," Thermochim. Acta, in press.

Heat Flow and Segregation in Directional Solidification

Massachusetts Institute of Technology
Professor A. F. Witt
NSG-7645

The research task is directed toward the optimization of crystal growth and segregation during solidification in Bridgman-type configurations. The first phase of this study was concerned with a determination of the effects of thermal boundary conditions on growth and segregation of doped Germanium in a conventional system. Making use of interface demarcation and spreading resistance analyses, it was found that at constant ampoule lowering rates, both growth and segregation remain non-steady state for growth lengths of up to 6 cm. The rate of growth is significantly less than the lowering rate under high axial thermal gradient conditions but exceeds the lowering rate by a factor of two at low applied thermal gradients. Upon temporary arrest of ampoule lowering uncontrolled growth or back melting takes place depending on the magnitude of the existing axial thermal gradient.

The segregation behavior observed is largely unexpected and as yet unexplained. It is found that during temporary arrests (periods of uncontrolled growth) the concentration of incorporated dopant (Ga) increases under conditions of decreasing rate of growth. Concentration maxima (up to 30 percent in excess of that for steady state growth) are reproducibly observed under a vanishing growth rate, while with the resumption of controlled growth the initial dopant concentration decreases under increasing rate of growth and fails to exhibit the expected value of $C_L k_0$.

The growth and segregation data obtained are of theoretical interest, but constitute primarily data input for a comprehensive thermal modeling approach to the design of a Bridgman-type growth system. The objective of currently conducted comprehensive thermal analysis is to obtain a quantification of the effects of furnace-imposed boundary conditions on system design based on the use of differential heat pipes separated by an adiabatic linear thermal gradient region.

Publications

Wang, C., Carruthers, J. R., and Witt, A. F., "Transient Crystal Growth and Segregation Behavior in Vertical Bridgman Configuration," in preparation.

Bourret, E. D., Favier, J. J., and Witt, A. F., "Theoretical Analysis of Directional Melting," submitted to Journal of Crystal Growth, 1981.

Vapor Phase of PbSnTe

Jet Propulsion Laboratory
Dr. J. A. Zoutendyk
NAS7-100 \$97K/year
March 1, 1980 - March 1, 1981

This effort consists of various studies of the effects of gravity-driven convection on the growth of PbSnTe from the vapor as a definition study for a possible flight experiment.

Publications

Zoutendyk, J. A. and Akutagawa, W. A., "Mass Transport in the Physical Vapor Growth of PbTe, SnTe and Their Alloys. I. Results for PbTe," submitted to Journal of Crystal Growth, January 1981.

B. SOLIDIFICATION OF METALS, ALLOYS, AND COMPOSITES

Solutal Convection During Directional Solidification

National Bureau of Standards

Dr. S. R. Coriell

R. J. Schaefer

H-27954H 160K/year

April 1977 - continuing task

See Section A, page 14, for full description of program task.

Liquid Phase Miscibility Gap Materials

S. H. Gelles Associates

Dr. S. H. Gelles

Dr. A. J. Markworth - Battelle Columbus Laboratories

NAS8-32952 Total Cost: \$480K

April 1978 - April 1983

The overall objective of the investigation is to determine the manner in which the microstructural features of liquid-phase miscibility gap alloys develop. The results of such a determination should make it possible to control the microstructures and the resultant properties of these alloys. The long-duration low gravity afforded by the Shuttle will allow experiments supporting this research to be conducted with minimal interference from buoyancy effects and gravitationally driven convection currents.

Ground based studies on Al-In, Cu-Pb, and Te-Tl alloys are presently being conducted to determine the effect of cooling rate, composition and interfacial energies on the phase separation and solidification processes that influence the development of the microstructure in these alloys. Both isothermal and directional cooling experiments are being conducted. Some understanding of the influence of gravity will be deduced by conducting these ground-based experiments at different angles to the gravitational vector. The ground-based experiment will be used as a technological base from which the flight experiments will be formulated and with which such flight experiments can be compared and judged. At the present time, isothermal experiments in the Al-In and Te-Tl systems and gradient freeze experiments in the Al-In system are contemplated for the Materials Experiment Assembly (MEA) to be flown on an early Shuttle flight. Isothermal, directional solidification, and containerless experiments are being planned for later Shuttle flights.

Publications

Gelles, S. H. and Markworth, A. J., "Microgravity Studies in the Liquid Phase Immiscible System: Aluminum-Indium," AIAA Journal, 16, 431 (1978).

Gelles, S. H., Bhat, B. N., et al., "Phase Separation in Transparent Liquid-Liquid Miscibility Gap Systems," Workshop on NASA Fluids Experiments System, University of Alabama, Huntsville, July 11-12, 1979, pp. 2-17.

Markworth, A. J., Gelles, S. H., et al., "Separation Kinetics of Immiscible Liquids in a Low-Gravity Environment," Nuclear Met. 20, 1023 (1978).

Gelles, S. H. and Markworth, A. J., "Agglomeration of Immiscible Liquids, Experiment 74-30," SPAR V Final Report, NASA TM-78275, August 1980.

Dendritic Solidification at Small Supercoolings

Rensselaer Polytechnic Institute

Dr. M. E. Glicksman

NAS8-32425 \$119K

March 1, 1977 - December 15, 1980

The objectives of this research task are to obtain information relating to the kinetic and morphological behavior of systems solidifying at small supercoolings, especially regarding the role of convective and diffusive transport and the influence of gravity. These studies provide important data on the fundamentals of solidification at normal terrestrial and reduced gravitational levels. Morphological features of interest include dendritic tip radii, sidebranch evolution, dynamic and isothermal coarsening. Kinetic features encompass axial growth rate as functions of spatial orientation, supercooling, and solute concentration.

The large data base now established for high-purity succinonitrile (SCN) had permitted the most comprehensive check of diffusional dendritic growth theory and the development of "scaling laws" permitting the extension of these theories to many other material systems. The current thrust of this program is on alloys based on SCN as the solvent (melt) system. Current efforts are focussed on SCN-argon, which models a dilute binary alloy. Other binary systems will be explored to establish the generality of these findings and their independence from any specificity inherent to many binary alloys. Finally, techniques are being developed and evaluated for measuring melt flow velocities adjacent to simple (cylindrical) and complex (dendritic) solid-liquid interfaces.

Publications

Glicksman, M. E., "Dendritic Solidification - Myths, Models, Measurements," The Metal Science Club of New York, April 7, 1977.

Glicksman, M. E., "Capillary Phenomena During Solidification," 5th International Conference on Crystal Growth, Cambridge, Massachusetts, July 19, 1977.

Glicksman, M. E., "Convective Heat Transfer During Dendritic Solidification," 16th Aerospace Sciences Meeting, Huntsville, Alabama, January 16-18, 1978, AIAA Paper 78-220.

Glicksman, M. E., "Advances in Solidification Fundamentals," 16th George Sachs Seminar on Advances in Foundry Technology, ASM Chapter, Syracuse, New York, April 11, 1978.

Glicksman, M. E., "Melting and Solidification: Microscopic Considerations and Single Phase Systems," ASM Symposium on Melting and Solidification of Metals: Theory and Practice, General Electric R&D Center, Schenectady, New York, May 23, 1978.

Glicksman, M. E., "Gravity-Induced Convection During Dendritic Solidification," COSPAR XXI Plenary Meeting, Innsbruck, Austria, June 1978.

Glicksman, M. E., "Influence of Spatial Orientation on the Kinetics of Convective-Diffusive Dendritic Growth," Fourth American Conference on Crystal Growth, NBS, Washington, D.C., July 18, 1978.

Glicksman, M. E., "Convective Heat Transfer During Dendritic Growth," 15th Meeting of Society of Engineering Science, Gainesville, Florida, December 4-6, 1978.

Glicksman, M. E., "Convection and Diffusion Effects During Dendritic Solidification," 17th Aerospace Sciences Meeting, New Orleans, LA, January 15-17, 1979.

Glicksman, M. E., "Development of 'Scaling Laws' and Their Application to Understanding Microstructural Evolution During Solidification," Petrie Lecture, Materials Engineering Department, Technion, Haifa, May 18, 1979.

Glicksman, M. E., "Convective Flow During Dendritic Growth," Fluids Experiments System Workshop, NASA/MSFC, Huntsville Alabama, July 12, 1979.

Glicksman, M. E., "Scaling Laws in Solidification," Connecticut Chapter of ASME, Wallingford, Connecticut, October 11, 1979.

Glicksman, M. E., "Influence of Metal Convection on the Kinetics of Dendritic Solidification," Olin Metals Research Laboratory, New Haven, Connecticut, October 12, 1979.

Glicksman, M. E., "Convection and Solidification Processes," Department of Mechanical and Aerospace Engineering Colloquium, Case Western Reserve University, November 14, 1979.

Glicksman, M. E., "The Role of Diffusive and Convective Transport in Solidification," Seminar lecture at the Chemical Engineering Department, Princeton University, November 1979.

Glicksman, M. E., "Recent Advances in Dendritic Growth Theory and Experiment," Seminar lecture at the E.I. DuPont Central Research Laboratories, Wilmington, DE, November 30, 1979.

Glicksman, M. E., "Transition from Diffusive to Convective Transport During Dendritic Solidification - A Basis for Future Experimentation at Reduced Gravity," 18th Aerospace Sciences Meeting, Pasadena, Ca, January 14, 1980.

Glicksman, M. E., "Dendritic Growth at Small Supercoolings," NASA Management and a Peer Panel at Marshall Space Flight Center, Huntsville, Alabama, March 5, 1980.

Glicksman, M. E., "Scaling Laws in Solidification," Metallurgy Division Colloquium, NBS, Washington, D.C., March 7, 1980.

Glicksman, M. E., "Relationships of Microstructure and Kinetics to Supercooling and Cooling Rate," 2nd International Conference on Rapid Solidification, Reston, Va, March 24, 1980.

Glicksman, M. E., "Dendritic Growth at Small Supercoolings," Seminar to the staff of Ames Laboratory, Iowa State University, Ames, IA, May 9, 1980.

Glicksman, M. E., "Boundary Layer Analysis for the Convection/Diffusion Transition in Dendritic Growth," Symposium on Theoretical and Applied Mechanics in Materials Sciences in Space, COSPAR XXIII Plenary Meeting, Budapest, Hungary, June 1980.

Glicksman, M. E., "Advances in Solidification Theory," Tutorial lecture to the Institut für Metallkunde und Werkstoffprüfung, Montanuniversität, Leoben, Austria, June 19, 1980.

Glicksman, M. E., "Quantitative Kinetic and Morphological Studies Using Model Systems," Conference on Modeling of Casting and Welding Processes, August 3-8, 1980, sponsored by the Engineering Foundation, Franklin Pierce College, Rindge, NH.

Glicksman, M. E., "Kinetics and Morphology of Dendritic Solidification," Seminar at Metallurgy & Materials Science Department, Case Western Reserve University, October 12, 1980.

Glicksman, M. E., "Role of Gravity in Solidification Processing," Seminar presentation, TRW MPS Industrial Committee, Redondo Beach, Ca, November 6, 1980.

Glicksman, M. E. and Huang, S. C., "Relationships of Microstructure and Kinetics to Supercoolings and Cooling Rate," in Rapid Solidification Processing, Principles and Technologies II, edited by R. Mehrabian, E. H. Kear, and M. Cohen, Claitor's Publishing Division, Baton Rouge, La, 1980.

Glicksman, M. E. and Huang, S. C., "Fundamentals of Dendritic Solidification, Part I - Steady State Tip Growth," Acta Met., 1981, in press.

Glicksman, M. E. and Huang, S. C., "Fundamentals of Dendritic Solidification, Part II - Development of Sidebranch Structure," Acta Met., 1981, in press.

The Influence of Gravity on the Solidification of Monotectic Alloys

Michigan Technological University

Dr. A. Hellawell

NAS8-33727 Total Cost: \$26K

September 1980 - September 1983

The objective of this research is to examine the monotectic reaction using directional solidification methods in order to obtain aligned composite structures. One aspect of the problem is the separation of two liquids below a miscibility gap and their incorporation within a duplex growth front. Both surface tensions and relative densities influence this process, and the objective of this research program is to identify the gravitational influence.

The systems under examination include Al-In, Cu-Pb, Al-Bi, Cd-Ga, and a transparent analogue $(\text{CH}_2\text{CN})_2\text{-H}_2\text{O}$ as well as the ternary systems Al-In-Sn, Cu-Pb-Al and Cd-Ga-Al. The transparent analogue system is being examined in a temperature gradient stage on an optical microscope in order to study the detailed form of the duplex, solid + liquid growth front. The particular objective here will be to look at the incorporation or rejection of liquid droplets at the growth front and to modify the behavior with ternary additions.

Publications

Hellawell, A. and Grugel, R., Proceedings of Third Conference on In-Situ Composites, Boston, Massachusetts, 1978, pp. 39-48.

Grugel, R. and Hellawell, A., "Alloy Solidification in Systems Containing a Liquid Miscibility Gap," Met. Trans. A. **12A**, 669-681 (April 1981).

Directional Solidification of Liquid Miscibility Gap Materials

Marshall Space Flight Center
Dr. M. H. Johnston
In-House

The objective of this program is to identify the influence of gravity on the aligned structure in liquid miscibility gap materials. This includes establishing the true monotectic composition and determining the solidification mechanism's possible dependence on undercooling.

Al-Bi and Al-Pb monotectic and off-monotectic materials will be solidified isothermally and unidirectionally. DTA measurements will be made to determine the true monotectic composition. Special attention will be given to the occurrence of undercooling prior to phase separation. Correlation between quantitative composition and melting point/undercooling data will be made in order to establish the solidification sequence.

Progress to date has included: (1) studying coarsening of rod structure; (2) ascertaining continuity of rods through etching techniques; observe buildup of constituents prior to aligned structure; (3) initiating contract with Vanderbilt University for differential thermal analysis, and (4) substantiating (through phase diagram studies) the possibility of undercooling in these systems.

Publications

Johnston, M. H. and Parr, R. A., "Directional Solidification of Aligned Monotectic and Hyper-Monotectic Materials," Proceedings of Conference on In-Situ Composites III, Materials Research Society, Ginn Custom Publishing, Lexington, MA.

Parr, R. A. and Johnston, M. H., "Growth Parameters for Aligned Microstructures in Directionally Solidified Aluminum-Bismuth Monotectic," Met. Trans. 9A, 1825 (1978).

Comparative Alloy Solidification

Marshall Space Flight Center
Dr. M. H. Johnston
In-House Cost: N/A

This effort is an extension of the earlier SPAR experiments, which used transparent model systems to investigate the gravitational influence on the solidification process, to actual metallic systems. Effects such as macro and micro segregation, grain size, shape, orientation, and physical properties of ingots cast in low-g will be compared to identical castings in one and high-g environments. A striking decrease in grain size with increasing g-field has already been demonstrated, confirming earlier predictions that dendrite multiplication was influenced by gravity-driven convective flows.

In the low-gravity solidification of the Sn-15wt%Pb alloy, the grain orientations were found to be completely isotropic, indicating probable nucleation in the center of the molten liquid. A significant increase in dendrite arm spacing was noted for the low-g metal sample, thus substantiating earlier results from the metal model systems.

A casting furnace has been completed and flown on SPAR VII and IX. This will be complemented by additional rapid-quench casting experiments carried out in KC-135 aircraft. A special furnace for this purpose has been constructed and is being tested for use beginning in mid CY 1980. The low-temperature SPAR furnace will also be flown to study the effects of partial solidification during low-gravity. Additional experiments could also be carried out in the drop tower when it is reactivated in mid CY 1980.

Publications

Johnston, M. H. and Griner, C. S., "The Direct Observation of Solidification as a Function of Gravity Level," Met. Trans A **8A**, 77-82 (1977).

Johnston, M. H., Griner, C. S., et al., "The Direct Observation of Unidirectional Solidification as a Function of Gravity Level," J. Cryst. Growth **50**, 831-838 (1980).

Johnston, M. H. and Griner, C. S., "The Direct Observation of Dendrite Remelting and Macrosegregation in Casting," SPAR V Final Report, NASA TM-78275, August 1980.

Studies of Model Immiscible Systems

Marshall Space Flight Center

Dr. L. L. Lacy

In-House \$50K/year

October 1, 1979 - October 1, 1982

The objectives of this program task are to use model organic immiscible systems to obtain fundamental information applicable to two-phase systems in general and to apply this understanding to materials of interest in the Materials Processing in Space program in order to interpret results of flight experiments involving monotectic alloys.

A number of model immiscible systems have been investigated. The systems, diethylene glycol/ethyl salicylate and cyclohexane/methanol have been purified and characterized in terms of phase diagrams and surface activity. Droplet migration in thermal gradients in the diethylene glycol/ethyl salicylate system has been demonstrated using small dimensional cells to suppress convective flows. The results of these preliminary experiments have been compared to theory with very favorable results. A report on these results is being prepared. More refined experiments are being initiated. Such measurements will also be conducted on KC-135 flights.

Solidification phenomena in two-phase miscibility gap systems have been initiated. The succinonitrile/water system is being used for these studies. Stokes flows are minimized by incorporating D₂O in appropriate quantities to equalize densities of the two phases. The effect of initial composition on the homogeneity of binary solids frozen through the miscibility gaps will be investigated.

A holographic microscopy system is being used to study growth, coalescence, and particle motions. Particle size distribution changes with respect to time and temperature are being determined from sequential holograms. The system is capable of working with particle densities up to 10^7 particles/cm³ and can resolve particles of the order of 2 to 3 μ in diameter throughout the entire cell volume.

Publications

Ross, S. and Nishioka, G., "Foams: Proceedings of Symposium Organized by the Society of Chemical Industry, September 1975," Academic Press, London, 1976.

Lacy, L. L., Nishioka, G. M., and Ross, S., "Investigations on Transparent Liquid Miscibility Gap Systems," Proceedings of Fluids Experiments System (FES) Workshop, UAH, July 1979, pp. 21-35.

Nishioka, G. M., Lacy, L. L., and Facemire, B. R., "The Gibbs Surface Excess in Binary Miscibility Gap Systems," J. Colloid Interface Sci. 80, 197-207 (March 1981).

Aligned Magnetic Composites

Grumman Aerospace Corporation
Dr. D. J. Larson Jr.
NAS8-32948 Total Cost: \$652K
July 1978 - July 1983

See Section A, page 25, for full description of program task.

Interfacial Destabilization in Metal Alloys

Laboratoire d'Etudes de la Solidification

Centre d'Etudes Nucleaires de Grenoble

Y. Malmejac

J. J. Favier

No Funds Exchanged

January 1980 - continuing task

The overall objectives of this investigation are to study the destabilizing mechanisms that affect a crystal growth interface, to obtain information on destabilized morphologies in the steady and transient states, and on growth kinetics behavior, and to attempt to separate the influences of liquid phase instabilities from the interface instability. These effects will be studied by directional solidification experiments on metal alloys with moderate melting temperatures under three generic types of conditions: (1) solidification at various rates with a given value of the temperature gradient in the melt next to the freezing interface, (2) solidification at a constant rate with the thermal gradient in the liquid next to the interface varying linearly with time; and (3) solidification with a constant thermal gradient in the liquid and solidification rates that vary linearly with time.

Ground-based research and experiment technique development will be performed to prepare for subsequent similar space experiments if the decision is made to proceed with the space phase of the investigation. As presently conceived, the space experiments will directionally solidify Sn-Bi (0.01 to 10 At.% Bi) and Bi-Sn (.01 to 10 At.% Sn) alloy samples at rates of 0.0001 to 0.1 cm/sec with liquid temperature gradients of 10 to 500 C/cm under weightless conditions. Samples will be solidified in simultaneous groups with one sample serving as a stationary reference interface so that a differential thermoelectric voltage generated by a moving interface may be continuously monitored under the quiescent conditions of space and related to the solidification morphology and velocity. Steady state and transient growth kinetics will be studied in relation to the thermal, compositional, and fluid flow effects.

Publications

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Lemaignan, C. and Malmejac, Y., "Interest and Difficulties of 0-g Studies of the Mechanisms of Eutectic Growth," 19th Meeting of the Committee on Space Research, Philadelphia, June 1976.

Favier, J. J. and Malmejac, Y., "The Contribution of Microgravity Experiments to the Study of Solidification Mechanisms," SPACELAB, Materials Research and Processing, London, November 1977.

Favier, J. J., "The Effect of Heat Exchanges on the Solidification Rate in a Thermal Gradient Furnace," 16th Aerospace Sciences Meeting, Huntsville, Alabama, January 1978, AIAA Paper 78-271.

Favier, J. J., "The Thermal Investigation of Solidification Carried Out in a Cartridge Furnace in Space," COSPAR XXI Meeting, Innsbruck, Austria, October 1978, pp. 535-543.

Favier, J. J., "Initial Transient Segregation During Unidirectional Solidification of a Binary Alloy in a Furnace with Thermal Damping," J. Cryst. Growth 49, 373-380 (1980).

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Favier, J. J., "Transient Growth Effects During Space Solidification Experiments in a Bridgman Configuration," COSPAR XXIII Meeting, Budapest, Hungary, June 1980, pp. 133-155.

Malmejac, Y., "European Present and Future Scientific Activities in the Field of Low Gravity Research," COSPAR XXIII Meeting, Budapest, Hungary, June 1980, pp. 61-64.

Wolf, H. S. and Malmejac, Y., "Microgravity Research in the Space Environment," ESA Bulletin, August 1980.

Analytical Approach to Modeling of Heat Flow in Bridgman-Type
Crystal Growth

Marshall Space Flight Center

Dr. R. J. Naumann

Ms. Ernestine Cothran

In-House

October 1980 - May 1981

See Section A, page 32, for full description of program task.

Directional Solidification of Magnetic Composites

Grumman Aerospace Corporation
Dr. R. G. Pirich
NAS8-32219 Total Cost: \$295K
February 1, 1977 - March 1981

Following the intriguing results obtained on the ASTP experiment, in which Mn-Bi eutectic directionally solidified in space was observed to have a finer microstructure and enhanced magnetic properties, extensive ground-based and flight investigation has been initiated. This has resulted in several significant findings.

Morphological analyses on eutectic Bi/MnBi samples that were directionally solidified during the 240-s low-g interval of the SPAR VI flight experiment show statistically smaller interrod spacings and rod diameters with respect to samples grown under identical solidification furnace conditions, in the same apparatus, in 1-g. The magnetic property measurements indicate that the flight samples contain ~7 v/o less dispersed MnBi than similarly processed 1-g samples for the same starting composition. Convectively driven temperature fluctuations in the melt, which result in unsteady liquid-solid interface movement in 1-g, are suggested to explain the morphological change between low-g and 1-g solidification. As a result of these fluctuations, an adjustment between the interrod spacing, growth velocity, and total undercooling at the solidification interface is proposed to account for the observed change in volume fraction of dispersed MnBi. Future low-g experiments involving both eutectic (SPAR IX) and off-eutectic (SPAR X) compositions are planned to quantify these unusual low-g effects.

Publications

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Larson, D. J. Pirich, R. G., et al., "The Influence of Partial Mixing on Directionally Solidified Bi/MnBi Magnetic Composites," Fifth Conference on Crystal Growth, Fallen Leaf, California, May 1980.

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Pirich, R. G., "Processing of Permanent Magnet Composite Materials," Annual Report of Francis Bitter National Magnet Laboratory, MIT Press, October 1979.

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- Pirich, R. G. and Adler, P. N., "Use of Magnetization to Characterize Transformation in Eutectic Bi/MnBi," TMS-AIME Fall Meeting, Pittsburgh, PA, October 1980.
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- Pirich, R. G., "Space Processing of Magnetic Composite Materials," Annual Report of the Francis Bitter National Magnet Laboratory, MIT Press, December 1980.
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Foam Copper

Marvalaud, Inc.

Professor R. B. Pond

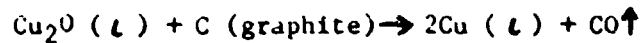
J. M. Winter

NAS8-33021 \$40K/year

April 27, 1978 - October 15, 1980

In one "g" gas bubbles move so quickly out of molten metals that it is not feasible to solidify a true "foam." In microgravity such density-driven flow can be insignificant. It may be feasible, therefore, to entrap gas bubbles during solidification in microgravity, resulting in a metal "foam." The objective of this work is to implement a microgravity experiment to determine if such an idea will work.

The idea is to use the reaction



to generate gas bubbles in a melt after it reaches microgravity. A deoxidized copper specimen is prepared with a homogeneous dispersion of fine graphite and a separate source of copper oxide. At 1100°C to 1150°C, only the graphite remains solid, serving as nucleation sites for gas bubbles. The whole experiment is contained at a positive pressure which is appropriate for the desired bubbles size.

Liquid Metal Diffusion in Solubility Gap Materials

Marvalaud, Inc.

Professor R. B. Pond

J. M. Winter

NAS8-33-46 \$34K/year

April 27, 1978 - September 15, 1980

The ultimate objective of this program is to measure the diffusion rates of two liquid metals. The intermediate objective is to verify or disprove the investigators' suspicion that determining diffusion constants of solubility gap liquid metals in one "g" experiments will lead to erroneous results due to density-driven convection motion. If this is true, the determinations will have to be done in microgravity. The immediate objective of this program is to conduct ground-based determinations of diffusion constants of the molten lead-zinc couple (at temperatures above and below the consolute) using utmost ingenuity to sort out (or suppress) the effects of gravity.

A new diffusion geometry has been devised which permits establishing a sharp, oxide-free, and void-free interface between the diffusion couples at a well-defined time after the melts equilibrate at a selected temperature. After allowing appropriate intervals for interdiffusion, the couples are quenched and analyzed by methods of quantitative metallography, quantitative chemical analysis, and x-ray fluorescence. The experiment permits having "g" in any selected direction.

Directional Solidification of Monotectic and Hypermonotectic
Aluminum-Indium Alloys under μ -g

Centre d'Etudes Nucleaires de Grenoble
Dr. C. Potard
No Funds Exchanged
September 1976 - present

The objective of this program is to analyze the mechanisms involved in the composite solid structure formation obtained from a miscibility gap alloy under microgravity. The metallic system aluminum-indium has been chosen for its low critical temperature, broad miscibility gap, and rather well-known thermodynamic properties.

Solidification of 10 and 50 atomic percent In samples will be carried out under a directional gradient in a SPAR rocket. Previous isothermal experiments by Gelles with this system resulted in almost complete phase separation in low-g within the In-rich material surrounding a core of Al-rich material. The mechanisms responsible for this separation are not understood, but because In preferentially wetted the alumina crucible, it is believed that capillarity may play an important role in the phase separation. The present experiment will use a SiC crucible which is preferentially wetted by the Al. The directional gradient will be used to investigate the effects of the droplet migration resulting from differential variations of the interfacial energy with temperature. Four experiments are required to obtain sufficient information to sort out the various effects.

The supporting ground-based research consists of: (1) Al-In phase diagram re-determination in the experiment composition range, (2) wetting properties of the two-phase liquid against silicon-carbide, (3) Earth gravity orientation effect on solid structures, (4) preliminary observations on capillary forces and coalescence, and (5) liquid-liquid and liquid-solid phase transformation studies (undercooling, kinetics, volume change).

Publications

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Potard, C., "SPAR Experiment 76-51: Experiment Readiness Review," May 18-19, 1978.

Boissier, J., Dusserre, P., and Potard, C., "Tests de Vibration et Vibration-Choc des Cartouches Prototypes SPAR," Internal Report, 1978.

Potard, C., "Preliminary Thermal Tests of the SPAR 76-51 Cartridges," Internal Report, 1978.

Potard, C., "A Review of the Mechanisms Involved in the Directional Solidification of Al-In Emulsions at Zero-Gravity," J. Brit. Interplan. Soc., Vol. 31, 1978, pp. 275-280.

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Potard, C., "Structures de Solidification des Alliages Immiscible Al-In et Al-Cd," Ann. Chim. Fr. 5, 279-287 (1980). June 19-21, 1979.

Direct Observation of Interface Stability

Stanford University

Professor W. A. Tiller

Professor R. S. Feigelson

Dr. Dennis Elwell

NAS3-33110 Total Cost: \$265K

December 1, 1978 - January 31, 1982

See Section A, page 40, for full description of program task.

C. FLUIDS, TRANSPORTS, AND CHEMICAL PROCESSES

Automated Analytical Electrophoresis Apparatus

University of Arizona - Tucson
Dr. Peter H. Bartels
NAS8-31948 Total Cost: \$300K
June 4, 1976 - August 1980

The objectives of this program were to provide an automated apparatus to determine and display the mobilities of living cells suspended in a compatible buffer and to establish its capabilities as an instrument for research and clinical usage.

The rationale was to provide two types of data: histograms of mobilities and a statistical evaluation of the cell populations. Histograms may reveal a specific component of a cell population in a pathological specimen. Histograms also give prior notice of abnormal mobility distribution of the sample; this enables the search to be confined to abnormal subpopulations. Manual determination of electrophoretic mobilities is unsuitable for this purpose in that it lacks the ability to make a large number of precise measurements in a short period of time.

Research tasks included designing all the components and interfaces; building and testing a prototype to demonstrate design feasibility; commencing measurements of cell populations and monitoring apparatus performance parameters; and correcting deficiencies noted including modifying apparatus as appropriate. The preceding tasks were completed to date. Measurements can be made faster than required. Limitations are photometric noise, accuracy of location determination and distance migrated at desirable voltages at selected microscope magnification. Nevertheless, 1000 measurements per sample can be made within 10 minutes, with an optimum cell concentration of 4×10^6 cells/cm³ with 3 to 5 lock-ons per cell.

Publications

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Brooks, D. E., et al., "Design and Testing of a Computer Controlled System (AEMS)," Cell Electrophoresis: Clinical Applications and Methodology, INSERM Symposium, Bristol, UK, 1979, pp. 409-419.

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Olson, G. B., Bartels, P. H., et al., "Computer-controlled Electrophoresis Microscope," SPIE 232, 54-61 (1980).

Bartels, P. H., Bartels, H. G., et al., "Automated Analytical Electrophoresis Microscope," accepted for publication in Cell Biophysics, 1981.

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Bartels, P. H. and Bartels, H. G., "Statistical Evaluation of Electrophoretic Mobility Measurements in the AEMS," Electrophoresis '81, Proceedings of First International Conference Electrophoresis Society, Charleston, SC, April 1981.

Hormone Purification by Isoelectric Focusing in Space

University of Arizona - Tucson
Dr. Milan Bier
NAS8-32950 \$85k/year
March 1978 - March 1982

The objectives of this research are to study the effects of gravity on the isoelectric focusing process, define and produce a definite isoelectric focusing experiment, and to refine future isoelectric focusing technology.

An inclusive list of gravitational hypothesis on gravitation effects will be generated. The hypothesis will be based on theoretical and experimental experience of the research team. Consideration will be given to resolution, throughput, boundary layers, convection, and sedimentation. In addition, tests using the previously developed Recycling Isoelectric Focusing (RIEF) apparatus will be made to verify aspects of each hypothesis using standard samples. A study will be conducted to define a meaningful space experiment in isoelectric focusing. Definition of the rationale, general approach, general configuration, and primary hardware requirements will be generated. Another study will refine the current mathematical model for steady state focusing of trace quantity proteins. Conductivity sensors will be designed, constructed, and incorporated into the current hardware.

Publications

Egen, N. B., Twitty, G. E., and Bier, M., "Recycling Isoelectric Focusing with Computer Controlled Data Acquisition System," 17th Aerospace Sciences Meeting, New Orleans, La., January 15, 1979, AIAA Paper 79-0405.

Bier, M. and Egen, N. B., "Large Scale Recycling Electro-focusing," in Electrofocusing/78, Hagland, Westerfield and Ball, Eds., Elsevier/North Holland, New York, 1979.

Bier, M., Egen, N. B., et al., "New Developments in Isoelectric Focusing," in Peptides, E. Gross and J. Meienhofer, Eds, Pierce Chemical Co., Rockford, Ill., 1979.

Bier, M. and Allyger, T. T., "Isotachophoresis," in Electrokinetic Separations Methods, P. G. Righetti, C. J. Van Oss, and J. W. Vanderhoff, Eds., Elsevier/North Holland, New York, 1979.

Palusinski, O. A., Allyger, T. T., et al., "Mathematical Modeling and Computer Simulation of Isoelectric Focusing with Electrochemically Defined Ampholytes, Biophysical Chemistry, in press.

Mosher, R. A., Palusinski, O. A., and Bier, M., "Computer Simulation and Experimental Validation of Isoelectric Focusing in Ampholine-Free Systems," submitted to J. Chromatography, 1981.

Palusinski, O. A., Bier, M., and Saville, D. A., "Mathematical Model for Transient Isoelectric Focusing of Simple Ampholytes," ready for submission.

Countercurrent Distribution of Biological Cells

University of Oregon Health Sciences Center

Dr. D. E. Brooks

NAS8-33575 Total Cost: \$226K

November 16, 1979 - November 15, 1982

The objectives of this research program are to develop and understand cell partition in a reduced gravity environment as a sensitive, analytical and high resolution preparative procedure for biomedical research.

In a reduced gravity environment the two-polymer phase will not separate via density-driven settling in an acceptably short length of time. It is to be expected that a certain amount of phase separation will take place, however, driven by the reduction in free energy gained when the interfacial area is reduced. This stage of the separation process will therefore depend directly on the magnitude of the interfacial tension between the phases. In order to induce complete phase separation in a short time, we are investigating electric field-induced separation which occurs because the droplets of one phase in the other have high electrophoretic mobilities which increase with droplet size. These mobilities are significant only in the presence of certain salts, particularly phosphates. The presence of such salts, in turn, has a strong effect on the cell partition behavior in dextran-poly(ethylene glycol) systems. The addition of the salts necessary to produce phase drop mobilities has a large effect on the interfacial tensions in the systems.

The other area in which work has been done is the further development of field-induced phase separation. We have been following phase separation in an electric field turbidimetrically. It has been found that phase separation, as indicated by optical clearing, occurs rapidly under the influence of a modest electric field but that turbidity then reappears after a few minutes. Apparently re-mixing or an instability in the phase system sets in after the initial separation. Current investigations involve making observations and trying to eliminate all possible artifacts from the measurements. These studies are hampered by the necessity of working with thin films of phase systems to avoid convective disturbance which make direct observation of the systems difficult.

Publications

Bamberger, S., Seaman, G.V.F., and Brooks, D. E., "Studies on the Interfacial Tension of Dextran/PEG System," Second International Conference on Partitioning, Sheffield, England, April 1981 (abstracts).

Brooks, D. E., Sorenson, P., and Van Alstine, J., "Detection of a Lipid Phase Transition by Partitioning of Liposomes," Second International Conference on Partitioning, Sheffield, England, April 1981 (abstracts).

Analysis of the Float Zone Process

Massachusetts Institute of Technology
Professor R. A. Brown
NSG-7645/Supplement 1

See Section A, page 10, for full description of program task.

Solutal Convection During Directional Solidification

National Bureau of Standards

Dr. S. R. Coriell

R. J. Schaefer

H-27954H 160K/year

April 1977 - continuing task

See Section A, page 14, for full description of program task.

Thermocapillary Flows and Their Stability: Effects of Surface
Layers and Contamination

Northwestern University

Dr. S. H. Davis

Dr. G. M. Homsy, Stanford University

NAS8-33881 Total Cost: \$400K

June 1980 - June 1983

The proposed research concerns the theoretical analysis of the fluid mechanics and heat transfer of motions driven by surface-tension gradients. The object is an understanding of the convection accompanying the process of growing high-quality crystals in a μ -g environment. The geometries considered included thin films, deep films and float-zone configurations. The particular aspects addressed are (1) the effects on steady Marangoni flow of contamination and the placement of third-phase films on the melt-gas interface, (2) the prediction of possible instabilities of Marangoni flows of pure melts, and (3) the effects on such instability criteria of contamination and surface films.

Calculation of flow and heat transfer characteristics is possible in the following limits: (1) $A \rightarrow 0$, (2) $M \rightarrow 0$, (3) $C \rightarrow 0$. These asymptotic limits, together with perturbation theory, may be used to analytically describe features of the structure of solutions to this class of problems. These analyses will be used to probe the effects of large classes of insoluble surface films on the steady flows generated by surface-tension gradients. It should be possible to describe what film characteristics are required to either suppress convection entirely, retard it to a given degree or even possibly augment it. The types of effects attributable to contamination would be determined. The magnitude of such effects could be calculated given experimental values for certain appropriate physical constants describing the material response of the contaminants.

Aggregation of Red Cells

University of Sydney
Dr. Leopold Dintenfass
MPS77F113

The objectives of this program are: (1) to determine whether the size of red cell aggregates, kinetics and morphology of these aggregates are influenced by near-zero gravity, (2) whether viscosity, especially at low shear rate, is afflicted by near-zero gravity (the latter preventing sedimentation of red cells), (3) whether the actual shape of red cells changes, and (4) whether blood samples obtained from different donors (normal and patients suffering from different disorders) react in the same manner to near-zero gravity. These are objectives for the first mission. Subsequent orbital flights intend to elaborate this data and introduce effects of plasma proteins, lipids, drugs, and various agents, in order to develop new diagnostic techniques and to obtain better insight into molecular aspects of blood rheology. These subsequent missions will depend in their construction on information obtained from the first flight.

It is possible that such data, obtained under near-zero gravity, when compared with equivalent laboratory data and subsequent procedures could form the basis for diagnostic tests. These subsequent procedures would encompass the response of blood samples or aggregates of red cells to the addition of drugs or agents which have various, even opposite, effects on the aggregation of red cells. Such agents or drugs will include fibrinogen, glucose, triglycerides, snake venom derivatives (i.e., Ancrod), beta blockers, etc. The kinetics of aggregation or disaggregation will be studied in parallel with the viscosity of blood. The results of these tests with compounds at different concentrations may well prove to be distinctive for blood samples from patients suffering from different diseases. It is possible that patients suffering from the same disease might exhibit different responses (in blood rheology) when subgrouped according to their ABO blood groups.

Publications

Dintenfass, L., "Aggregation of Red Cells and Blood Viscosity Under Near-Zero Gravity," Biorheology 16, 29-36 (1979).

Dintenfass, L., "Aggregation of Red Cells and Blood Viscosity Under Zero and One Gravities - Instrumentation for Spacelab 3," Aust. Phys. Sci. Med. 2, 437-451 (1979).

Dintenfass, L., "Aggregation of Red Cells and Blood Viscosity as Models of Materials Experimentation in the Space Environment," 17th Aerospace Sciences Meeting, New Orleans, La., January 15-17, 1979.

Dintenfass, L. and Jedrejzyk, H., "Morphology and Kinetics of Aggregation of Red Cells in 12.5 Micrometer Slits: A Micro- and Macro-Photographic Study," First European Haemorheology Conference, Nancy, France, 1979.

Dintenfass, L., "Kinetics and Morphology of Red Cells: Preparations for the NASA Spacelab," in Rheology, Volume 3: Applications, Plenum Press, New York, 1980.

Dintenfass, L., "Haemorheology and the Microcirculation: Role of the Inversion Phenomenon," in Progress in Microcirculation Research, D. Garlick, Ed., Committee on Postgraduate Medical Education, University of Sydney, 1980.

Dintenfass, L., "Haemorheology of Capillary Perfusion," in Capillary Perfusion, Chapter IV, R. M. Hardaway, Ed., 1980.

Dintenfass, L., "The Clinical Impact of the Newer Research in Blood Rheology," Angiology, 1981.

Transient Thermal Convection in Low-g

NASA Headquarters

Dr. R. F. Dressler

In-House

January 1980 - continuing

The purpose of this research program is to obtain analytical solutions for transient and periodic convection flows for arbitrary low-g excitations with imposed thermal gradient in cylinders and cubes, for both 2-D and 3-D flows.

Publications

Dressler, R. F., "Transient Thermal Convection During Orbital Spaceflight," in press, 1981.

Dressler, R. F., "Approximate Analysis of Transient Convection in the Lal MPS Cell," work completed, manuscript in preparation.

Surface Tensions and Their Variations with Temperature and Impurities

National Bureau of Standards
S. C. Hardy
H-27954B \$200K/year
April 1977 - continuing task

In this program traditional sessile drop surface tension measurements are being used in conjunction with Auger spectroscopy and other modern surface analytic techniques to study the thermodynamics and chemistry of liquid metal interfaces. The materials selected for study are generally, but not exclusively, those of interest in other areas of the space research program. In recent work, for instance, the surface tension of liquid gallium was measured as a function of temperature from the melting point to about 750°C. The temperature dependence of the surface tension was found to be approximately quadratic. This data is being used elsewhere in the design of experiments which will use gallium to study Marangoni flow.

Currently we are working on the application of Auger spectroscopy to liquid metal surfaces. The experiments are being conducted in a conventional Auger spectrometer with a vertical cylindrical mirror analyzer and a horizontal sample manipulator. The samples are in the form of sessile drops which permits the surface tension to be measured simultaneously with the Auger spectrum. Initial work with gallium drops has been promising because we found that the surface of the drop can be cleaned by sputtering with argon ions. Fluid flows are generated in the sputtering which draw solid impurities such as oxides into the ion beam where they are sputtered away. The mechanism generating this fluid flow is not yet identified. At present we are studying the segregation of tin at the surface of gallium-tin alloys.

Publications

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Hardy, S. C., "The Motion of Bubbles in a Vertical Temperature Gradient," J. Colloid & Interface Sci., Vol. 69, March 1979, pp. 157-162.

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New Polymers for Low-Gravity Purification of Cells by Phase Partitioning

University of Alabama in Huntsville
Dr. J. Milton Harris
NAS8-33978 \$47k/year
September 1, 1980 - August 31, 1982

A potentially powerful technique for separating different biological cell types is based on the partitioning of these cells between the immiscible aqueous phases formed by solution of certain polymers in water. This process is gravity-limited because cells sediment rather than associate with the phase most favored on the basis of cell-phase interactions. We are presently involved in the synthesis of new polymers both to aid in understanding the partitioning process and to improve the quality of separations. The prime driving force behind the design of these polymers is to produce materials which will aid in space experiments to separate important cell types and to study the partitioning process in the absence of gravity (i.e., in an equilibrium state).

Three new types of water-soluble polymers have been synthesized. These are: (1) polyethylene glycols with attached crown ethers; (2) polyethylene glycols with attached cyclodextrins; and (3) dextrans with attached long-chain hydrocarbons. The crown ethers and cyclodextrins are of interest because of their ability to selectively form complexes with, respectively, metal cations and hydrophobic anions. Consequently, these materials present the possibility of specifically binding groups on the cell surface. The dextrans with long-chain hydrocarbons attached are of interest because of the probable attraction of the hydrocarbon for lipophilic areas on the cell surface. Testing of the properties of these new materials has begun.

Purification and Cultivation of Human Pituitary Growth Hormone-Secreting Cells

Pennsylvania State University

Dr. W. C. Hymer

NAS9-15566 \$65k/year

June 1980 - June 1981

Human growth hormone (hGH) is in demand for treatment of pituitary disease, osteoporosis, stress ulcers, etc. However, the demand is far greater than available supply. The problem could be solved by the culture of purified hGH cells that produce hormone. This program addresses the problem of (a) separation of the pituitary growth hormone cell, (b) its maintenance in vitro, and (c) assessment of the role that gravity plays in establishing limits of these current laboratory technologies.

In an effort to fulfill the above objectives a human pituitary column perfusion method has been developed to sustain hGH release from pituitary tissue over extended periods (1-3 days). On the basis of experimental results from 144 human pituitary glands removed 1-18 hours, postmortem, it has been found that prostaglandin E_1 ($10^{-9}M$) or epinephrine (10^{-9}) stimulates release of a "GRF" from rat hypothalmi which is, in turn, capable of sustaining hGH release for at least 24 hours. Tissue samples stained immunocytochemically for hGH cells reveal large numbers of well-preserved cells in this experimental protocol. These results support the notion that the human postmortem pituitary gland contains functional growth hormone cells.

Results from numerous experiments demonstrate that we can prepare 15×10^3 cells/mg postmortem human pituitary tissues. These cell preparations are 80% viable, and by electron microscopy contain membrane and granule systems characteristic of intact tissue. Concerted efforts have been made to separate GH cells from both rat and human pituitaries by chemistry gradient electrophoresis. Results indicate that somatotrophs (GH cells) apparently have low electrophoretic mobilities, and possibilities for their eventual purification by this technique appear encouraging. Finally, methodology has been developed for the implantation of human pituitary cells in rats. With this methodology it should be possible to assess function of hGH cells in vitro, as well as eventually being able to isolate hGH from the animal.

Publications

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Hymer, W. C., Wilbur, D. L., et al., "The Human Post Mortem Pituitary Contains Functional Somatotrophs," 62nd Meeting of the Endocrine Society, Washington, D.C., June 1980.

Hymer, W. C., Wilbur, D. L., Page, R., et al., "Pituitary Hollow Fibers Units In Vivo and In Vitro," Neuroendocrinology, in press, 1981.

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Hymer, W. C. and Wilbur D. L., "Pituitary Cell Systems In Vitro and In Vivo," in Functional Correlates of Hormone Receptors, in press, 1981.

Hymer, W. C. and Todd, P., "Purification of Rat Pituitary Somatotrophs by Density Gradient Electrophoresis," submitted to Cell Biophysics, 1981.

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Biosynthesis/Separations Laboratory-Development of a Space
Biosynthesis System and Biological Studies for Electrophoresis
in Space

Johnson Space Center
Dr. Dennis R. Morrison
Mr. Bernard J. Mieszkuc
In-center \$120k/year
January 1981 - continuing task

The objectives of this program are to: (1) obtain data on the performance of cell culture vessel system elements and to define the biological oxidation process--the transfer of oxygen from gas to liquid and from liquid to oxidant, and (2) determine the limits of ground-based technology using a preprototype reactor for studying enzymatic reactions and suspension cell cultures.

The Biosynthesis/Separations Laboratory supports the Materials Processing in Space studies on biosynthesis and cell separations for investigations into the production of high value pharmaceuticals which are very difficult or impossible to obtain on Earth with currently available technology. This laboratory is responsible for the biological science supporting the Electrophoresis Equipment Verification Test scheduled to fly on STS-3 and subsequent electrophoresis experiments under micro-gravity conditions.

The laboratory has both monolayer and suspension cell culture capabilities. Current research includes procedures for the obtaining of cell cultures, and the freezing and storage of cells. Procedures for growing cell cultures in suspension are being investigated. A continuous line of baby hamster kidney cells has been grown in suspension, and the growth of cells on microcarriers is being pursued. A variety of beads were used as substrates for the attachment of cells. Procedures for the analysis of biochemicals produced by cell cultures have been established. Fibrinolytic and colorimetric methods are being used routinely for the assay of urokinase. The production of urokinase in monolayers of human embryonic kidney cells has been demonstrated. Biochemical purification of secreted products on affinity columns is being developed. Procedures for the chromosome analysis of cell cultures (counting and karyotyping) have been established. Continuing efforts include: (1) the design, construction, verification testing, and flight test of a small space bioreactor to demonstrate the advantages of these new techniques using mammalian cells in culture, (2) the identification of requirements and hardware design concepts for small cell incubators needed to maintain living cells on board Shuttle or Spacelab before and after continuous flow electrophoresis experiments in conjunction with the NASA Joint Endeavor Agreement with McDonnell Douglas Astronautics Corporation, and (3) the screening and selection of living cells to be separated in future space flight experiments.

Surface Tension-Driven Convection Phenomena

Case Western Reserve University
Dr. Simon Ostrach
Dr. Adin Mann
NAS8-33015 \$226K/year
May 1978 - August 1981

A combined theoretical and experimental program is being conducted to determine the nature and extent of motions induced by surface-tension gradients along fluid interfaces. This research is aimed at both giving greater understanding of containerless processing techniques and developing theoretical and experimental methods to achieve that goal.

Generally in problems involving free-surfaces the surface shape must be determined as part of the solution. This makes analyses extremely complex. Thus, the complete free-surface boundary conditions including effects of surface curvature and deformation were formulated and the proper dimensionless parameters are being derived. In this way the importance of interface shape changes can be readily assessed. Problems of increasing complexity are being studied to determine the most effective and efficient means of solving such problems.

Two specific experimental investigations are in progress. The first is a study of surface-tension gradient convection in a deep liquid layer. The objectives are to obtain basic velocity and temperature fields, to develop experimental techniques, and to test various fluids for their suitability for such experiments. The second experiment concerns the flow fields in a liquid column which is suspended vertically between two ends that can rotate and be at different temperatures. In this simulated float zone the flow and temperature fields due to the interaction between surface-tension gradient and rotational driving forces can be investigated. Other heating modes, such as ring heater, will also be examined.

Publications

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Lowry, S., "An Experimental Study of Heat Induced Surface-Tension Driven Flow," MS Thesis, Case Western Reserve University, Department of Mechanical & Aerospace Engineering, Cleveland, Ohio, 1980.

Electrophoretic Separation Based On Immunomicrospheres

Jet Propulsion Laboratory
Dr. A. Rembaum
In-Center \$100k/year
October 1978 - continuing task

The objective of this task is to (1) demonstrate a new concept for cell separation based on labeling specific groups of cells with immunomicrospheres and isolating the labeled cells and unlabeled cells by means of electrophoresis and (2) to demonstrate that the cell separation of immunologically labeled cells is more efficient in the space environment than Earth. By proper choice of positively and negatively charged microspheres, the electrophoretic mobility (μ) may be altered to achieve separation of cells hitherto impossible to achieve.

Free flow electrophoresis is a powerful method of separating large numbers of cells. The present project is designed to eliminate the main drawbacks of the technique, i.e., loss of resolution due to gravity-induced convection currents, caused by Joule heating, and insufficient electrophoretic mobility differences between the desired and undesired cell populations. By performing electrophoresis on-board orbiting spacecraft, gravity-induced convection currents are eliminated. Therefore, experiments are planned to test this hypothesis. The immunomicrosphere technology is applied to solve the problem of insufficient mobility differences. Immunomicrospheres are submicron, functionalized, polymeric particles coated with antibodies or other reagents capable of recognizing and binding to individual cell types. By choosing microspheres of a mobility at least 20% lower than that of the target cell, we have been able to electrophoretically separate human B and T lymphocytes, a separation which is impossible without immunomicrospheres. This type of separation should be considerably improved in the absence of gravity.

Publications

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Rembaum, A. and Margel, S., "Design of Polymeric Immunomicrospheres for Cell Labeling and Cell Separation," British Polymer Journal 10, 275 (1978).

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Margel, S., Zisblatt, S., and Rembaum, A., "Polyglutaraldehyde: A New Reagent for Coupling Proteins to Microspheres and for Labeling Cell Surface Receptors. Part II: Simplified Labeling Method by Means of Nonmagnetic and Magnetic Polyglutaraldehyde Microspheres," J. Immuno. Methods 28, 341 (1979).

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Mass Transfer in Electrolytic Systems Under Low Gravity
Conditions

University of Alabama in Huntsville

Dr. C. Riley

Dr. H. D. Coble

Dr. R. B. Owens, MSFC

Gordon Fisher, INCO

NAS8-33542 \$8K

September 1979 - May 1980

Electrodeposition involves mass transfer from one phase to another. In particular one is concerned with the deposition of materials into the solid phase out of the liquid (solution or suspension) phase. Diffusive and convective flow coupled with deposition result in density gradients that are gravity dependent. We desire to characterize this gravity dependence associated with electrodeposition. When a better understanding of the gravity influence is acquired, it should lead to improved control of variables during electroformation of materials. The overall objectives will be the electroformation of materials with improved or more desirable properties and a better understanding of the transport of inert suspensions during the electrodeposition process.

Miniature electrodeposition cells have been constructed. Simple metal-in/metal-out reactions using cobalt and copper are being utilized as the mass transfer agents. The density flow patterns between electrodes with both a vertical and horizontal configuration will be bench characterized using both laser Schlieren and interferometry detection. The metal-in/metal-out systems will obviate gas evolution interference on the observation system. These results will be compared to those determined for the same cells under reduced gravity conditions produced during parabolic, free-fall flights of a KC-135 aircraft. Studies with neutral buoyancy particles will be used to model the transport of neutrals under low gravity conditions.

Publications

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Fluid Dynamics of Crystallization from Vapors

University of Utah - Salt Lake City

Dr. F. Rosenberger

NSG-1534 Total Cost: \$271K

June 1, 1978 - May 31, 1981

See Section A, page 35, for full description of program task.

Mathematical Models of Continuous Flow Electrophoresis

Princeton University

Dr. D. A. Saville

Dr. R. S. Snyder, MSFC

NAS8-32614 Total Cost: \$202K

August 1977 - January 1982

Development of high-resolution continuous flow electrophoresis devices ultimately requires comprehensive understanding of the ways various phenomena and processes facilitate or hinder separation. A comprehensive model of the actual three-dimensional flow, temperature and electric fields shall be developed to provide guidance in the design of electrophoresis chambers for specific tasks and means of interpreting test data on a given chamber.

Part of the process of model development shall include experimental and theoretical studies of hydrodynamic stability. This is necessary to understand the origin of mixing flows observed with wide-gap gravitational effects; the suppression of gravity may allow other processes to become important.

To insure that the model accurately reflects the flow field and particle motion requires extensive experimental work. Much of the experimental work can be done under terrestrial conditions if the roles of gravity are appreciated and taken into account properly. Even though the resolution of a terrestrial-based machine may be unsatisfactory, verification of the model will provide the support necessary for the interpretation of microgravity operations. Recommendations shall be made for the design and operations of the ground experiments.

Publications

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Saville, D. A. and Lynch, E. D., "Heat Transfer in the Thermal Entrance Region of an Internally Heated Flow," Chemical Engineering Communications, in press.

Electrophoresis Technology

Marshall Space Flight Center

Dr. R. S. Snyder

In-House \$60K/year

The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation, (2) characterize and optimize electrophoretic separators and their operational parameters, and (3) separate biological cells using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separation.

The following results have been accomplished: (1) experiments have been designed to decouple or minimize the fluid effects due to the flow process, electrokinetic effects, and temperature gradients, (2) transparent electrophoresis chambers have been built allowing measurement of internal and wall temperature while observing flow perturbations, (3) techniques have been developed to map the temperature and flow fields in the chamber with small disturbance to the process, (4) the sensitivity of these chambers to lateral temperature gradients has been measured and a new, all-metal chamber has been designed to incorporate the improvements suggested by these experiments, (5) analysis has yielded results that reproduce flow distortions observed in experimental chambers, (6) the DESAGA FF48 and Beckman continuous flow electrophoresis chambers have been compared using standard particles (fixed red blood cells) under various operating conditions. Optimum operating parameters for resolution and throughput have been established and the two devices can be compared, and (7) these optimized conditions are being used for the separation of biological cells and macromolecules with reproducibility.

Publications

Rhodes, P. H., "Sample Stream Distortion Modeled in Continuous Flow Electrophoresis," NASA TM-78178, August 1979, 45 pgs.

Snyder, R. S. and McGuire, J. K., "Characterization of Continuous Flow Electrophoresis for Improving Resolution and Throughput," submitted to Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981.

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Rhodes, P. H. and Snyder, R. S., "Numerical Analysis of Continuous Flow Electrophoresis," submitted to Electrophoresis '81, Walter De Gruyter & Co., Berlin, 1981.

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McGuire, J. K., Miller, T. Y., et al., "New Experimental Approaches to the Isoelectric Fractionation of Cells," J. Chromato. 194, 323-333 (1980).

Fluid Dynamics Numerical Analysis

Lockheed Missiles & Space Company
Dr. L. W. Spradley
Dr. J. Robertson
NASW-3281 Total Cost: \$200K
August 1979 - August 1981

The purpose of this research program is to compute transient thermal convection for cases of importance to Materials Processing in Space. This includes problems too difficult for analytical solutions and also includes verification of ranges of validity of theory developed by Dr. R. F. Dressler.

Lockheed's previously derived GIM code has been modified and adapted for these tasks. Work is completed on two-dimensional transients for stepfunctions for circles and squares. Ranges of validity of Rayleigh number for the Dressler theory have been determined. Axisymmetric transient convection in a sphere idealizing the Lal experiment has been completed. Several cases have been completed simulating gravity vector shifts in two-dimensional cylinders. The three-dimensional flow cases have not yet begun.

Publications

Robertson, S. J. and Spradley, L. W. "Numerical Analysis of Natural Convection in Two-Dimensional Square and Circular Containers in Low Gravity," 23rd Meeting of Div. of Fluid Dynamics, American Physical Society, Ithaca, N.Y., November 1980.

Physical Phenomena in Containerless Glass Processing

Clarkson College of Technology

Dr. R. Shankar Subramanian

Dr. Robert Cole

NAS8-32944 Total Cost: \$554K

December 1977 - December 1982

The objective of this work is to study the behavior of bubbles inside drops of model fluids and molten glasses in free fall, focusing on their migration and interaction. Such migration will be induced by thermocapillarity, rotation and/or oscillation of the drop. The results of the experiments are expected to be of use in the development of techniques for mixing and fining glasses in space and in providing a better understanding of how microballoons are formed.

A broad ground-based investigation into the various physical phenomena of importance in the space experiments is under way. Theoretical models of thermocapillary flow in drops, thermal migration of bubbles in drops, and the migration of bubbles in rotating liquid bodies are being developed. Experiments have been conducted on the migration of a bubble to the axis of a rotating liquid body and the rise of bubbles in molten glass. Experiments on thermocapillary motion in pendant drops, the influence of oscillation on bubble migration, and volatilization phenomena in glasses are under way.

Publications

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Wilcox, W. R., Subramanian, R. S., et al., "Screening of Liquids for Thermocapillary Bubble Movement," AIAA Journal 17, 1022-1024 (1979).

Cole, R., Papazian, J. M., et al., "Bubble Departure Radii of Solidification Interfaces," Int. J. Heat Mass Transfer 23, 219-224 (1980).

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Subramanian, R. S. and Cole, R., "Glass Processing in Space," Workshop on Containerless Processing, General Electric Space Center, Valley Forge, Pennsylvania, October 1980.

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Kidney Cell Electrophoresis

Pennsylvania State University

Dr. Paul Todd

NAS9-15584 \$40K/year

June 1980 - continuing task

The objective of this investigation is to repeat the MA-011 experiment under conditions which are optimum for the viability of human kidney cells and most favorable for the best possible electrophoretic separation of those few (about 5%) cells which produce urokinase or human granulocyte conditioning factor (HGCF), and erythropoietin.

This study effort will perform the ground-based research necessary to establish all of the optimum experimental conditions required to accomplish the best possible electrophoretic separation of human kidney cell fractions, which produce urokinase, granulocyte stimulating factor, or erythropoietin. This overall effort will include: (1) development of optimum buffer systems, (2) viability tests, (3) ground-based research on electrophoretic mobilities, (4) development of standard cell culture methods and assays for urokinase, granulocyte stimulating factor, and erythropoietin, and (5) acquisition of the ground control data to be compared with results using cells returned from the electrophoretic separations carried out in microgravity.

It has been found in studies conducted on cells from 16 explants that electrophoretic heterogeneity exists in early-passage cultures and that cells from intermediate-mobility fractions appear to be most efficient in urokinase production. In tests of the flight buffer "A-1" it has been found that DMSO is superior to glycerol for cell freezing and that the low ionic strength required for electrophoresis is not a significant factor in determining cell viability. It has also been found that there is considerable variability among cell preparations from different explants, so criteria have been established for the selection of cells for microgravity experiments.

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Production of Large-Particle-Size Monodisperse Latexes in Microgravity

Lehigh University

Dr. J. W. Vanderhoff

Dr. F. J. Micale

Dr. M. S. El-Aasser

NAS8-32951 Total Cost: \$418K

February 22, 1978 - February 22, 1983

The effort will explore the possibility of performing seeded polymerization in low-g to avoid the problems of creaming and sedimentation as the particles grow and change density. If successful, this could provide a method for routinely producing monodisperse spheres in a size range that is difficult to obtain by other techniques.

Ground-based experiments are continuing to obtain kinetic data and to evaluate alternative methods for growing such spheres. Flight hardware has been defined, and negotiations are under way to produce reactors to fly on early Shuttle flights.

Work has continued with the high swelling ratio of monomer to polymer technique developed in the laboratory. Emphasis has recently been put on the evaluation of newly obtained mercaptans which are used in the pretreatment of latex seed. A correlation is being sought between the molecular weight distribution, as a function of the type and concentration of mercaptan, and the swelling ratio. Work has also been carried out, in a continuing effort, toward an evaluation of the degree of agitation obtained in the laboratory prototype monodisperse latex reactor, LUMLR, as a function of rpm.

Publications

Vanderhoff, J. W., et al., "Heterogeneous Chemical Reactions: Preparations of Monodisperse Latexes," Final Report, NAS8-32399, July 1977.

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D. ULTRAHIGH VACUUM AND CONTAINERLESS
PROCESSING TECHNOLOGIES

Thermophysical Properties of Refractory Materials at High Temperatures

National Bureau of Standards

J. H. Colwell

A. Cezairliyan

L. A. Schmid

H-27954B \$100K/year

April 1978 - continuing task

The objective of this research is to develop the containerless techniques required for the determination of the thermophysical properties of refractory materials at high temperatures.

The approach being pursued in this study will utilize the low-gravity space environment to maintain a spherical sample within a cold-walled frequency field to hold the sample in position. The sample will be heated to a high temperature by inductive and/or radiant heating and then allowed to freely cool by radiation alone. As the sample cools, its surface temperature and total radiant energy loss will be measured radiometrically as a function of time, giving a determination of the heat capacity of the sample. Temperature gradients within the samples will complicate the interpretation of many experiments, but this can be circumvented by measuring samples of different size and may even permit the thermal conductivity of the material to be determined under certain conditions. The limiting parameters of these measurements are being determined by computer modelling experiments. Laboratory experiments are being conducted with the various detectors to establish the required measurement techniques.

Publications

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Ultimate Intrinsic Coercivity SmCo₅ Magnet

Charles Stark Draper Laboratory

Dr. Dilip K. Das

Dr. R. T. Frost, General Electric

NAS8-33607 Total Cost: \$396K

September 24, 1979 - September 23, 1982

The objective of the proposed research is to produce Sm-Co magnets of reasonably high maximum energy product with intrinsic coercivity. These magnets will have to be composed of extremely fine, defect-free crystallites. A very large amount of oxygen enters the magnet during the fabrication process, which involves (1) cold compaction of magnetically aligned powder, (2) comminution of the alloy to fine particle size powder, and (3) thermal densification of the cold compact. The normal practice is to perform the first two steps in air, and the thermal densification is produced by sintering in a purified noble gas atmosphere.

Design is now in progress to build a chamber where controlled atmosphere comminution and cold compaction of aligned powder will be performed. While still in the chamber the cold compact will be sealed into an outgassed and evacuated steel cannister by heli-arc welding, before removing from the chamber. The steel cannister with the magnetic compact inside will then be subjected to hot isostatic processing (HIP'ing). The HIP'ing procedure for SmCo₅ developed at the CSDL, is now at an advanced stage and will be readily applicable to the NASA magnetic program.

In preparation for the design of a high quality comminution and encapsulation chamber, some preliminary comminution experiments in a laminar flow nitrogen glove box have been performed. The oxygen pick-up in the above powder has been reduced by 60 percent over the powder prepared in air. Based on this encouraging result, using a high quality chamber in a pure quiescent noble gas should lower the oxygen content to a few decades of ppm instead of the 0.6 weight percent in the state-of-the-art magnets.

Publications

Das, D. K., Kumar, K., et al., "Ultimate Intrinsic Coercivity Samarium-Cobalt Magnet," Draper Laboratory, Monthly Progress Report, February 1981.

Das, D. and Kumar, K., "Hot Isostatically Pressed SmCo₅ Magnets," IEEE Trans. on Magnetics MAG-16, 1000-1002 (September 1980).

The Upgrading of Glass Microballoons

Bjorksten Research Laboratories

Dr. Stanley A. Dunn

Robert T. Nagler

Elmer G. Paquette

Stephen Gunter

Dr. E. J. Crosby

NAS8-33513 \$43.8K/year

August 28, 1978 (repropose each 12 months)

The objective of this program is to study extensively the processes and mechanisms involved in producing glass microballoons of acceptable quality for laser fusion by gas jet levitation and manipulation in the molten condition.

The gas supporting levitation microfurnace (LMF) concept represents a unique means of handling and shaping small bodies where contact with foreign condensed phase objects or supports could be deleterious. Levitation is achieved by aerodynamic lift imparted to the body from a gaseous flow field generated with the aid of a collimated hole structure (CHS). A long-standing objective of the development of the LMF has been the perfecting of glass microballoon (GMB) geometry by centrifugal centering and coordinated temperature and pressure manipulation. Centering by centrifugal means implies the ability to control the orientation of the axis of rotation as well as the speed.

Existing LMF designs provide excellent horizontal and vertical translational stability. A third requirement, that of rotational control has been partially solved. Rate of rotation can be controlled down to very slow speeds. The ability to stop rotation entirely and to index the GMB to any desired orientation are objectives now being sought through division of the field of jets into three or four regions, each with separately controllable gas flows. The capability of being able to stop rotation as well as to control position will be of use in studies of heat transfer involving GMB's.

Publications

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Homogeneous Crystallization Studies of Borderline Glass Forming Systems

Marshall Space Flight Center
Dr. E. C. Ethridge
Mr. J. R. Johnson
In-House \$60K/year
April 1, 1981 - April 1, 1984

The primary objective of this study is to use containerless as well as pseudocontainerless processing techniques to melt and resolidify borderline glassformers in such a way as to obtain critical cooling rates to avoid homogeneous crystallization. A secondary objective is to develop new techniques for supercooling oxide melts to produce bulk samples of candidate materials for ground-based screening tests of potential flight compositions.

The research plan is to melt and resolidify samples in containerless and pseudocontainerless fashion. For rapid cooling rates, pendant drop melting and rapid solidification in free fall will be utilized. For slower cooling rates, vitreous supports during sample heating and cooling will be used. Critical cooling rates and crystallization rates will be measured.

Rework of the SPAR Electromagnetic Levitator (EML) for Materials
Experiment Assembly (MEA) Accommodations

General Electric Company
Dr. R. T. Frost
NAS8-34231 \$117k/year
October 1980 - October 1981

The general goals of this project are to study the upgrade requirements and approaches needed for incorporation of an EML in the MEA carrier, to design and develop an engineering version of a multisample specimen exchanger, to develop a design concept for an automated calorimeter, and to carry out support tasks for the Electromagnetic Containerless Processing Task Team.

Publications

Frost, R. T. and Stockhoff, E. H., Minutes of the Electromagnetic Containerless Processing Task Team Meetings, Vailey Forge Space Center, April 20, 1979, and Marshall Space Flight Center, October 1979.

Wouch, G. and Frost, R. T., "Results of Containerless Solidification Experiments in the Terrestrial and Space Environments," presented at AIME Meeting, New Orleans, LA, February 21, 1979.

Racette, G. W. and Frost, R. T., "Deposition of Polycrystalline Silicon Films on Metal Substrates Under Ultra-High Vacuum," J. Cryst. Growth 47, 384-388 (1979).

Frost, R. T., Racette, G. W., et al., "Ultra Vacuum Epitaxial Growth of Silicon," 8th International Vacuum Congress, Cannes, France, September 1980.

Containerless Processing of Optical Glass in Space

Rockwell International

Ralph Happe

NAS8-32023 Total Cost: \$624K

April 1979 - April 1983

The overall program task is to increase the knowledge of containerless melting and cooling of oxides or other related inorganic compounds. It is expected that containerless melting and cooling will minimize the possibilities of heterogeneous crystal nucleation on cooling from the melt by eliminating such crystal nucleation sites as container walls and other crystalline material. Such suppression of heterogeneous nucleation should enhance glassforming possibilities and permit the preparation of glasses in bulk form from materials whose low viscosity prevents such preparation in massive form by terrestrial processes. Containerless processing also promises to permit the preparation of glasses from substrates whose melting points and/or reactivity is too high to permit melting in any known container material.

Terrestrial research has been and will continue to be performed, leading to flight experiments aboard sounding rockets (SPAR) and the Space Shuttle. The terrestrial research relies heavily on a technique of air suspension with CO₂ laser melting developed by the Principal Investigator to identify promising compositions for space research. Among such compositions identified to date are binary gallia-calcia and the higher melting alumina-calcia and lanthana-alumina-calcia compositions.

Following screening of candidate compositions, further terrestrial research will be performed with the most promising compositions to determine suitable processing techniques for preparing samples for space research aboard sounding rockets, early Space Shuttle flights and, in more sophisticated equipment, on later Shuttle/Spacelab flights.

Undercooling Studies in Metastable Peritectic Compounds

Marshall Space Flight Center
Dr. L. L. Lacy
In-House \$80K/year
March 1, 1979 - March 1, 1982

The objective of this program task is to investigate undercooling and containerless solidification of metastable superconducting alloys Nb_3Ge and Nb_3Al and pure metal melts such as Nb; specifically, to investigate the structure and superconducting properties of undercooled Nb-Ge alloys and to determine the feasibility of forming metastable Nb_3Ge in bulk form.

Pure Nb droplets have been undercooled in excess of 500 K in free fall using the MSFC drop tube. The droplets form single crystals with no shrink cavity in the interior. The outer surface is rough, indicating the shrinkage associated with solidification was taken up by the interdendritic fluid. NbGe droplets have also been deeply undercooled and rapidly solidified in the drop tube. The undercooling has been measured for the NbGe alloy drops with results showing that the Nb 18 a/o Ge drops undercooled 500 K, where the Nb 22 a/o Ge drops undercooled 300 K. These undercoolings do not represent the maximum extent possible since these drops undercool the complete length of the drop and nucleated only after reaching the catcher. An increase in the transition temperature of the heavily undercooled NbGe drops have a measured transition temperature of $\sim 10K$ which is 4K above the as cast materials. The increase indicates that at least some of the metastable AIS structure has been formed. Further work includes annealing SEM and TM analysis as well as X-Ray diffraction studies to determine the extent of formation of the metastable phase. Further drops are also planned with recently purchased material with a more stoichiometrically perfect composition.

Publications

Lacy, L. L., Robinson, M. B., and Rathz, T. J., "Drop Tube Experiments on Undercooled Niobium and Niobium Alloy Droplets," 108th AIME Meeting, New Orleans, LA, 1979.

Robinson, M. B., Rathz, T. J., et al., "Drop Tube Apparatus for Low-Gravity Solidification," NASA Tech Briefs, MFS-25242, 1980.

Nisen, N. B., Lacy, L. L., and Robinson, M. B., "Containerless High-Temperature Calorimeter," NASA Tech Briefs, Spring 1979, p. 89.

Steinberg, J., Lord, A. E., Lacy, L. L., et al., "Production of Bulk Amorphous $\text{Pd}_{77.5}\text{Si}_{16.5}\text{Cu}_6$ in Continuous Low-Gravity Environment," Appl. Phys. Lett. 38, 135-137 (February 1981).

Rathz, T. J., "Superconducting Measurements on Nb-Ge Alloys Using Susceptibility Techniques," MS Thesis, University of Alabama in Huntsville, 1980.

Robinson, M. B., "Undercooling Measurement in a Low Gravity Containerless Environment," MS Thesis, University of Alabama in Huntsville, 1981.

Lacy, L. L., Robinson, M. B., and Nisen, N. B., "Containerless High-Temperature Calorimeter," Patent #4,248,083, issued Spring, 1981.

Lacy, L. L., Rathz, T. J., and Robinson, M. B., "Containerless Undercooling and Solidification of Bulk Metastable Nb_3Ge Alloys," submitted to Journal of Applied Physics, May 1981.

Lacy, L. L., Rathz, T. J., and Robinson, M. B., "Containerless Undercooling and Solidification in Drop Tubes," J. Cryst. Growth 51, 47-61 (January 1981).

A Proposal to Measure the Viscosity of Molten Pd₇₈Si₁₆Cu₆
A Critical Experiment to Support Possible Space Flights

Drexel University
Dr. Arthur E. Lord
NAS8-33372 \$29K/year
February 1979 - June 1980

Metallic glasses are materials that are cooled very rapidly to bypass crystallization. Due to the lack of crystallinity, these materials have very unique properties (very "soft" magnetic properties, high hardness, high yield stress, corrosion resistance, etc.). In general, only small sizes are available. To ascertain the largest sizes that are theoretically possible to manufacture, the time-temperature-transformation (TTT) curves must be determined. The most important physical property needed for the TTT diagram calculation is the viscosity and its temperature dependence.

The viscosity of Pd₈₂Si₁₈ and Pd_{77.5}Si_{16.5}Cu₆ have also been determined. The viscosity of these alloys is of the order of 50 poise at their melting points--a value about 100 times that of any other known metal. Calculation using standard nucleation/growth/transformation theory may be able to produce amorphous Pd_{77.5}Si_{16.5}Cu₆ at cooling rates as low as 10°C/sec.

Publications

Anderson, P. M. and Lord, A. E., "Exact Values for Crystallization Temperatures at Different Heating Rates from Isothermal Transformation Times," Mat. Sci. Engr. 43, 93-96 (1980).

Anderson, P. M. and Lord, A. E., "Correlation Between Viscous Flow and DSC Measurements at the Glass Transition in the Metallic Glass Alloy Fe₄₀Ni₄₀P₁₄B₆," Mat. Sci. Engr. 43, 267-270 (1980).

Anderson, P. M. and Lord, A. E., "Viscosity of Metglas 2826 Near the Glass Transition Using Rapid Heating," J. Noncryst. Solids 37, 219-229 (1980).

Anderson, P. M. and Lord, A. E., et al., "Continuous Cooling (CT) Versus Isothermal Transformation (TTT) Diagrams in Metallic Alloy Glasses," J. Noncryst. Solids 34, 267-272 (1979).

Anderson, P. M. and Lord, A. E., "The Effects of Structural Relaxation and Crystallization on Isothermal Viscous Flow Near the Glass Temperature in Metglas 2826," Mat. Sci. Engr. 44, 279-284 (1980).

Anderson, P. M. and Lord, A. E., "The Effects of Structural Relaxation and Crystallization on Isothermal Viscous Flow Near the Glass Temperature in Metglas 2826," Mat. Sci. Engr. 44, 279-284 (1980).

Anderson, P. M. and Lord, A. E., "Effect of Hydrostatic Pressure on the Magnetic Properties of Certain Metallic Glasses," J. Appl. Phys. 48, 4839-4843 (1977).

Anderson, P. M. and Lord, A. E., "Demonstration of Phase Transition and Properties of Metallic Glasses for Undergraduates," Am. J. Phys. 46, 80-82 (January 1978).

Steinberg, J. and Lord, A. E., "Acoustic Emissions Generated at the Glass Transition in Silicate Glasses," J. Am. Ceram. Soc. 63, 234-235 (1980).

Steinberg, J., Tyagi, S., and Lord, A. E., "Effect of Thermal Annealing on the Thermal Expansion of Amorphous Alloys," Journal of Noncrystalline Solids (submitted June 1980).

Steinberg, J., Tyagi, S., and Lord, A. E., "Thermally Induced Acoustic Emission in Glasses," NDT International, October 1980, pp. 225-229.

Steinberg, J., Lord, A. E., et al., "Production of Bulk Amorphous $\text{Pd}_{77.5}\text{Si}_{16.5}\text{Cu}_6$ in a Containerless Low-Gravity Environment," Appl. Phys. Lett. 38, 135-137 (February 1981).

Steinberg, J., Tyagi, S., and Lord, A. E., "Calculation of the Critical Cooling Rate for Amorphous $\text{Pd}_{77.5}\text{Si}_{16.5}\text{Cu}_6$," Applied Physics Letters (submitted August 1980).

Tyagi, S., Steinberg, J., and Lord, A. E., "Anomalous Thermal Expansion in Amorphous $\text{Fe}_{78}\text{Mo}_2\text{B}_{20}$," Appl. Phys. Lett. 37, 618-620 (October 1980).

Wouch, G. and Lord, A. E., "Eddy Currents: Levitation, Metal Detectors, and Induction Heating," Am. J. Phys. 46, 464-466 (May 1978).

Lord, A. E. and Anderson, P. M., "Acoustic Emissions Generated During Viscous Flow of Metallic Glasses," Lett. Appl. Engr. Sci. 5, 489-494 (1979).

Lord, A. E., Wouch, G., and Frost, R. T., "Preparation of Amorphous Ferromagnetic Materials Through Containerless Solidification," SPAR IV Report, NASA TM-78235, January 1980.

Measurement of the Properties of Tungsten at High Temperatures

Rice University
Dr. J. Margrave
NAS8-33199 \$20K/year
November 1978 - March 1981

This research is directed toward the measurement of the thermophysical properties of tungsten using containerless techniques. The properties of tungsten are of interest because it lies at the extreme of metal melting points and is a key datum in any extrapolation or interpolation process. In addition, difficulties in handling molten tungsten may establish the limitations of ground-based containerless systems in processing materials at high temperatures.

Samples are suspended containerlessly by an electromagnetic levitator. Additional heat is supplied by electron bombardment. Temperatures are measured by pyrometers. Heat capacities are determined from cooling curves, and total enthalpy is obtained using a drop calorimeter.

Analysis of Degassing Techniques to Support Vacuum Research Facility (SVRF)

McDonnell-Douglas Astronautics Company

B. C. Moore

NAS8-33155 Total Cost: \$99.9K

16 months

The objectives of this program task were: (1) to develop data on cleanliness levels and outgassing techniques, (2) document present Aerospace technology and procedures in ultrahigh vacuum systems and high cleanliness spacecraft, (3) identify SVRF Degassing Methods, evaluate each method and select optimum, and (4) define scenarios for fabrication and operation of the SVRF.

Publications

Analysis of Degassing Techniques to Support Space Vacuum Research Facility, McDonnell-Douglas, NAS8-33155, Vols. IA, II, III, and V, September 11, 1979.

System Feasibility of a Space Vacuum Research Facility - Wake Shield Demonstration, McDonnell-Douglas, NAS8-33155, Vols. IB and IV, January 30, 1980.

Moore, B. C., "Proposed Method for Degassing Metals for Extreme Low Pressures," J. Vac. Sci. Technol. 7, (July-August 1980).

Ultrapure Glass Optical Waveguide Development in Microgravity by the Sol-Gel Process

Battelle Columbus Laboratories
Dr. S. P. Mukherjee
JPL 955361 Total Cost: \$74K
April 1980 - November 1980

The objectives of the program were to characterize the glasses obtained by the melting of the gels prepared by different preparation procedures. The high alkali compositions [SiO_2 60, B_2O_3 15, Na_2O 25 (weight percent)] were chosen for the development of the different gel preparation procedures. Three different preparation procedures using three sources of Na_2O were developed. The sources of Na_2O were sodium methylate, sodium nitrate, and sodium acetate. The influence of gelling process parameters such as the chemical nature of the reactants, the pH of the medium, the concentration of water and the constituent oxides were studied. The results show that the physio-chemical nature of the gels is strongly influenced by the process parameters. The rate of removal of residual organic groups from the gel during the thermal treatments below 500°C is related to the gel preparation procedures and the conditions of thermal treatment such as the humidity and the ambient atmosphere during thermal treatment and the rate of heating.

The gels prepared by two procedures using sodium methylate and sodium acetate were noncrystalline and no crystallinity developed on thermal treatment at 600°C for 40 hours. The gels prepared by procedures using sodium nitrate were partially crystalline due to the presence of residual sodium nitrate. Infrared spectroscopic studies of the gels prepared by the different procedure show that there exists some finer structural difference in the gels prepared by different procedures. This difference could be due to the difference in the polymeric structure of the gels originated by the different preparation procedures. Molecular structures of glasses obtained by melting the gels at a lower temperature ($\leq 1000^\circ\text{C}$) prepared by the different procedures were not identical. However, the melting at high temperatures ($\geq 1200^\circ\text{C}$) causes equilibrium to reach into the melts from the different gels.

Publications

Mukherjee, S. P., "Sol-Gel Processes in Glass Science and Technology," Proceedings of the International Conference on Frontiers of Glass Science, University of California, Los Angeles, July 16-18, 1980.

Mukherjee, S. P., "Chemistry of the Sol-Gel Process in the Alkali-Borosilicate System," presented at the American Ceramic Society Annual Meeting, Washington, D.C., May 1981.

Mukherjee, S. P. and Neilson, G. F., "The Microstructures and Small Angle X-Ray Scattering of Gel-Derived Glasses," presented at the American Ceramic Society Annual Meeting, Washington, D.C., May 1981.

Ultravacuum Vapor Epitaxial Growth of Silicon

General Electric Company

Dr. C. A. Neugebauer

NAS8-33121 Total Cost: \$105K

August 30, 1978 - March 31, 1980

See Section A, page 34, for full description of program task.

Glass Shell Manufacturing in Space

KMS Fusion

Dr. Robert L. Nolen

NAS8-33703 Total Cost: \$266K

December 6, 1978 - December 5, 1981

The principal objective of this research is to develop a detailed understanding of the chemical and physical processes involved in the formation of uniform, high-quality spherical glass shells. Where possible, various stages of the shell-blowing process will be formulated into mathematical models.

The temperature regimes where major transformations occur in the shell starting materials (metal-organic gels) have been identified. This was accomplished using a combination of thermal analytical techniques. The gases generated by pyrolysis of the gel were quantitatively characterized by gas chromatography and pressure tests.

A mathematical model of the heat transfer mechanisms occurring in the gel-to-shell transformation will be refined from one previously formulated under another contract. Computerization of the mathematical model will be used to bring it into line with the results from a series of controlled drop tower furnace experiments to be done with fully characterized and standardized gel powder pellets. Experiments conducted in the course of modeling the heat transfer process should also provide information on gas transport mechanisms. Also modeling will begin on uniformity of the wall thickness.

Publications

Nolen, R. L., "Glass Shell Manufacturing in Space," First-Fourth Quarterly Reports, NAS8-33703, March 1979-January 1980.

Downs, R., Ehner, M., Homyk, and Nolen, R. L., "Analysis of Shell Blowing Gases from Metal Organic Gels," J. Am. Vac. Soc. 18 2000 (1981).

Containerless High Temperature Property Measurements by Atomic Fluorescence

Yale University
Dr. P. C. Nordine
NAS8-34383 \$271K/year
June 1, 1980 - May 31, 1983

The objective of this program is to measure high temperature properties in containerless experiments using laser excited atomic fluorescence. Its purposes include: (1) the development of new techniques for Earth-based study of candidate Spacelab high temperature experiments and material processing applications and (2) the measurement of high temperature thermodynamic and transport properties. The method is to obtain absolute temperature and concentration profiles in the gaseous boundary layer surrounding a hot solid or liquid by laser excited atomic fluorescence measurements.

The basic idea is that a laser beam, whose bandwidth includes a doppler and pressure broadened atomic absorption line will, in the absence of quenching processes, produce fluorescence from that species at an intensity proportional to its concentration. Therefore, the spatial variation in fluorescence intensity from ambient or vaporizing species can be used to measure boundary layer temperature and concentration profiles. The experimental parameters which may be controlled (e.g., specimen dimensions and temperature, total pressure, uniform or nonuniform boundary layer temperature, etc.) allow experiments in which thermodynamic, transport and/or gas phase kinetics control the observable gradients. Thus, specimen vapor pressure, temperature, or evaporation rate, gas phase transport properties, or gas phase reaction rate constants may be determined.

Containerless experiments will be carried out by use of aerodynamic and/or electromagnetic levitation. Where the solution of boundary layer transport equations is necessary to the interpretation of results, fluorescence measurements on the stagnation line (in natural or forced convection) will be used to simplify the data analysis.

Publications

Nordine, P. C. and Atkins, R. M., "Aerodynamic Levitation of Laser Heated Solids in Gas Jets," submitted to Review of Scientific Instruments, 1981.

Electrostatic Control and Manipulation of Materials for Containerless Processing

Jet Propulsion Laboratory

Dr. M. M. Saffren

Dr. D. D. Elleman

Dr. W. K. Rhim

In-Center \$650K (to date)

October 1978 - continuing task

The objective of this task is to develop electric field positioning/manipulation techniques and technology for the containerless processing of materials in bulk and dispersed forms. This method obviates limitations of other methods. Two principal demonstrations to prove technique and technology will be completed no later than CY 83. An important part of the task is to ensure that facilities to be developed satisfy requirements of identified user applications. Another part of the task is to pursue study of the electrohydrodynamics of liquid drops (that must be done as part of the development) as a fundamental investigation.

Task studies include: electric field feedback control for bulk sample positioning, methods and limitations of electric field confinement, electrode configuration, electro-hydrodynamics of both charged and neutral drop systems, and dynamics and stability of charged particle arrays. These investigations are performed in the laboratory, in the KC-135 aircraft, and through theoretical and numerical study. Use is made of available SPAR flight experiment data, and Shuttle flight experiments are planned.

Publications

Saffren, M. M., Elleman, D. D. and Rhim, W. K., "Dynamics of Liquid Shells," Conference on Inertial Confinement Fusion, San Diego, California, February 26-28, 1980.

Saffren, M. M., Elleman, D. D., and Rhim, W. K., "Dynamics of Compound Liquid Drops," JPL Report 715-27: "Theoretical Development--Concentric Drops--Mode Frequencies and Interfacial Displacements," Vol. I; "Theoretical Development--Energy and Angular Momentum," Vol. II; "Numerical Results," Vol. III; "Non-Concentric Modes and Centering Forces--Theoretical Development," Vol. IV.

Saffren, M. M., Elleman, D. D., and Rhim, W. K., "Dynamics of Compound Drop Systems," International Colloquium on Drops and Bubbles, Monterey, CA, November 19-20, 1981.

Rhim, W. K., Saffren, M. M., and Elleman, D. D., "Electrostatic Levitation Devices at JPL," Materials Research Society Symposium, Boston, MA, November 16-18, 1981 (Submitted).

Physical Phenomena in Containerless Glass Processing

Clarkson College of Technology

Dr. R. S. Subramanian

Dr. Robert Cole

NAS8-32944 Total Cost: \$544K

December 1977 - December 1982

See Section C, page 106, for full description of program task.

Convection in Grain Refining

Massachusetts Institute of Technology
Professor J. Szekely
Professor M. C. Flemings
NSG-7645

The purpose of this program is to obtain a better understanding of the relationship between fluid flow phenomena, nucleation, and grain refinement in solidifying metals both in the presence and in the absence of a gravitational field. An ultimate technical aim is to determine ways to achieve significant grain size reductions in hard-to-process melts.

The work planned has the following components: (1) the computation of the electromagnetic force field, (2) computation of the fluid flow fields resulting from the electromagnetic force fields, (3) the verification of the calculations by experimental measurements, and (4) the interfacing of the computer results with the grain refining studies.

Experimental work in this program is aimed at establishing relations between melt supercooling, convection, and dendritic structure (including grain refinement). The bulk of this work has been conducted in a levitation-type apparatus. Supercoolings up to 270° have been obtained and summary results to date include: (1) a dendritic morphology with relatively coarse grain structure (typically between 100 and $1000\mu\text{m}$) is obtained for superheated samples and for samples undercooled less than 170°C , (2) some samples undercooled possess a spherical "non-dendritic" morphology with a grain size in the range of 10 to $50\mu\text{m}$ depending on initial undercooling and cooling rate after nucleation, and (4) undercooled samples show unusual segregation behavior including solute-rich cores, and solute-rich islands, the latter of which may form from remelting during recalescence.

Publications

Grevet, J., Szekely, J., and El-Kaddah, N., "An Experimental and Analytical Study of Gas Bubble Driven Circulation Systems," submitted to Journal of Heat and Mass Transfer, 1981.

Choudhary, M. and Szekely, J., "The Effect of Temperature Dependent Electrical Conductivity on Flow and Temperature Fields in Slags in ESR Systems," submitted to Metallurgical Transactions, 1981.

Crystal Nucleation in Glass-Forming Alloy and Pure Metal Melts
Under Containerless and Vibrationless Conditions

Harvard University
Professor David Turnbull
NAS8-32691 Total Cost: \$120K
June 23, 1978 - June 22, 1981

The main objective of this research is to characterize nucleation behavior in glass-forming alloy melts. Such experiments should indicate if formation of alloy glasses in bulk form is possible and, if so, what are the necessary conditions. The most favorable conditions would be those in containerless, vibrationless experiments in high vacua or inert atmospheres.

The crystal nucleation behavior of Au_4Si and Pd_4Si glass-forming melts and of pure Ni by the droplet technique is being investigated. It has been found that the onset undercooling, ΔT_0 , for copious nucleation in molten Au_4Si droplets varies widely with thermal treatments which alter the nature of the SiO_2 film on the droplet surface. However, ΔT_0 as large as 1/3 of the liquidus temperature for some droplets was observed. Glass and crystallization temperatures of Au_4Si based alloys are sharply increased ($\sim 1^\circ$ per atom %) when Cu replaces some of the Au.

Publications

Turnbull, D., "On Anomalous Prefactors from Analysis of Nucleation Rates," Progress in Materials, in press.

Spaepen, F. and Turnbull, D., "Negative Pressures and Melting Point Depression in Oxide-Coated Liquid Metal Droplets," Scripta Met. 13, 149 (1979).

Thompson, C. V. and Spaepen, F., "On the Approximation of the Free Energy Change on Crystallization," Acta Met. 27, 1855 (1979).

Fusion Target Technology

Jet Propulsion Laboratory

Dr. T. G. Wang

In-Center \$300K/year

October 1979 - continuing task

The objectives of this task are to (1) study the physical processes that are associated with the fabrication of inertial confinement fusion (ICF) targets in a weightless environment, (2) determine jointly with DOE centers the need for extended zero gravity in the future production of ICF targets, and (3) provide technological information to DOE centers that is pertinent to their current target fabrication research.

To produce the high-quality fusion target shells that are required, four fundamental physical processes must be understood: spheroidization of the shell, uniformity of shell thickness and coating, adiabatic expansion, and contraction of the molten pellet as it passes through temperature gradient environments.

The approach to be taken is to (1) study the fluid dynamic processes that pertain to pellet fabrication processes such as bubble centering, coating uniformity, and various instabilities; (2) study the effects of various temperature levels and temperature gradients on pellet fabrication; (3) construct Earth-based high temperature and high temperature gradient drop towers; (4) initiate development of a process for the studies, numerical analyses, and computer calculations on various pellet fabrication processes.

Publications

Wang, T. G., "Gravitational Effects on Target Fabrication," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Elleman, D. D., Croonquist, A. P., et al., "Low Gravity Acoustic Positioning Chamber for Liquid Shell Studies," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Lee, M. C., Helizon, R. S., et al., "Real-Time Image Acquisition and On-line Image Processing of a Hollow Sphere Confined in a Drop Furnace," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Kendall, J. M., "Fluid Dynamics Relating to the Production of Large Fusion Targets," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Advanced Containerless Processing Technology

Jet Propulsion Laboratory

Dr. T. G. Wang

In-Center \$350K/year

October 1978 - continuing task

The primary long-range objectives of this task are to: (1) study and advance the science of contactless positioning and manipulation of a high-temperature acoustic chamber, (2) provide design information on a flight version of this chamber for materials science studies in a contactless and zero gravitation environment, and (3) provide potential MPS investigators with a set of ground-based facilities with which to perform precursor experiments.

The approaches to be taken in FY 81 are experimental and theoretical studies of (1) acoustic positioning and manipulation capabilities of a rectangular chamber as a function of temperature and pressure, (2) various acoustical geometries which may have special application in materials science studies, (3) loss mechanisms associated with high-intensity and high-temperature acoustic waves, (4) an aeroacoustic positioning system which will allow us to levitate heavy samples in the laboratory, (5) a liquid-liquid positioning system which will allow us to study the dynamics of liquid melts, and (6) positioning and manipulation capabilities of a KC-135 acoustic module.

Publications

Lagomarsini, G. and Wang, T. G., "Acoustic Containerless Processing Module for Materials Research," 17th Aerospace Sciences Meeting, New Orleans, LA, January 1979, AIAA Paper 79-0369.

Jacobi, N., Tagg, R. P., et al., "Free Oscillations of a Large Drop in Space." 17th Aerospace Sciences Meeting, New Orleans, LA, January 1979, AIAA, 79-0225.

Lee, M. C., "Temperature Dependence of Molecular Vibrational Relaxation Frequency for O_2-H_2 , O_2-He and O_2-CO_2 Mixture Between 300-675°K," Proceedings of the Ultrasonics Symposium, IEEE, New Orleans, September 1979.

Barmatz, M. B., "Nonlinear Effects in a High Intensity Acoustic Resonant Chamber," Proceedings of the 10th International Congress on Acoustics, Sydney, Australia, July 1980.

Trinh, E., Wang, T. G., and Lee, M. C., "A Technique for the Study of Drop Dynamics in a Liquid-Liquid System," JASA 67, S1 (1980).

Wang, T. G., "Acoustic Levitation and Manipulation for Space Applications," Proceedings of the Ultrasonics Symposium, IEEE, New Orleans, LA, September 1979, pp. 471-475.

Wang, T. G., Elleman, D. D., et al., "Containerless Processing Technology Experiment Report, SPAR IV Experiment 76-20," SPAR IV Final Report, NASA TM-78235, January 1980.

Wang, T. G., Elleman, D. D., et al., "Containerless Processing Technology Experiment Report, SPAR VI Experiment 76-20," May 1981.

APPENDIX A
MPS ORGANIZATIONS

5 ORGANIZATIONS

Alabama A&M University
Huntsville, AL

Athens State College
Athens, AL

Battelle Columbus Laboratories
Columbus, OH

Bjorksten Research Laboratories
Madison, WI

Case Western Reserve University
Cleveland, OH

Charles Stark Draper Laboratory
Cambridge, MA

Clarkson College of Technology
Potsdam, NY

Drexel University
Philadelphia, PA

EG&G Corporation
Santa Barbara, CA

French Atomic Energy Commission
Nuclear Research Center of Grenoble
Grenoble Cedex, France

S. H. Gelles Associates
Columbus, OH

General Electric Company
Corporation Research Laboratories
Schenectady, NY

General Electric Company
Space Sciences Laboratories
Valley Forge, PA

Grumman Aerospace Corporation
Bethpage, NY

Harvard University
Cambridge, MA

NASA
Headquarters
Washington, D. C.

Honeywell Research Laboratories
Lexington, MA

Iowa State University
Ames Laboratory, ERDA
Ames, IA

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, CA

NASA
Johnson Space Center (JSC)

KMS Fusion, Inc.
Ann Arbor, MI

NASA
Langley Research Center (LaRC)
Hampton, VA

Lehigh University
Bethlehem, PA

A. D. Little, Inc.
Cambridge, MA

Lockheed Corporation
Huntsville Research & Engineering Center
Huntsville, AL

Marvalaud Corporation
Westminster, MD

Massachusetts Institute of Technology
Cambridge, MA

Michigan Technological University
Houghton, MI

NASA
George C. Marshall Space Flight Center (MSFC)
Marshall Space Flight Center, AL

McDonnell Douglas Corporation-West
Huntington Beach, CA

McDonnell Douglas Corporation-East
St. Louis, MO

National Bureau of Standards
U. S. Department of Commerce
Washington, D.C.

Northwestern University
Evanston, IL

Pennsylvania State University
University Park, PA

Princeton University
Princeton, NJ

Rensselaer Polytechnic Institute (RPI)
Troy, NY

Rice University
Houston, TX

Rockwell International
Downey, CA

Rockwell International
Thousand Oaks, CA

Semtec, Inc.
Huntsville, AL

Stanford University
Stanford, CA

University of Alabama, Huntsville (UAH)

University of Arizona
Tucson, AZ

University of Oregon
Health Sciences Center
Portland, OR

University of Sydney
Sydney, AUSTRALIA

University of Utah
Salt Lake City, UT

University of Wisconsin - Milwaukee

Universities Space Research Association
Columbia, MD

Yale University
New Haven, CT

APPENDIX B
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APPROVAL

MATERIALS PROCESSING IN SPACE PROGRAM TASKS

Compiled by E. Pentecost

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

for William C. Snoddy
J. E. KINGSBURY
Acting Director
Space Sciences Laboratory

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